Smith

Chapter 5 Organic Analysis
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Analysis of Ink by paper chromatography

Introduction

Paper chromatography was developed in the early 1940's in England. The technique is based on the fact that paper contains a thin film of water around the cellulose fibers of the paper, called a stationary phase. A mixture of the compounds to be separated is placed in a small spot at one end of a strip of paper, and an organic solvent (mobile phase) is passed over the spot and across the paper. Since each compound present has a different size, shape, and distribution of electrical field, each compound will dissolve in the water and organic solvent to a different extent.

The net result is that if two compounds are started at the same place and solvent passed over them, one compound will move along the paper faster than the other will. After a period of time the flow of the mobile phase is stopped. The paper is dried and then sprayed with a reagent that will produce colored spots. The material used in our experiment is already colored, so the latter step is not required.

Some years ago, the color in inks was made of a single component substance. Therefore, when ink was chromatogrphed, only one color spot was evident. Inks manufactured in more recent times are more often multi-component material, with the ink color due to a mixture of dyes. These inks, then, show a variety of colored spots when chromatographic separation is performed.

In this analysis, we will determine whether two different documents were written with the same ink, or at least inks produced during the same period of manufacturing technology.

Crime scene

Two documents are submitted to the document examination section of a forensic laboratory with the following explanation and request.

The patriarch of a very affluent family has written, in long hand, a statement giving one of his daughter's permission to invest a substantial sum of money in a business venture. The second document is a statement that instructs the daughter to confer with other members of the family concerning investments before they are undertaken. Both have been written with a fountain pen containing black ink. The daughter maintains the document was written at a much later time. You are to determine in the inks are the same or different. Preparation for chromatographing the ink samples involves the determination of a proper developing solvent.

Equipment

6 Thumbtacks	1 Test tube rack	6 corks, fitted with wire hooks	1pr Scissors
1 Graduated cylinder	1Spotter (capillary tube)	1pr goggles	2 Test tubes (10cm long)
1 Pencil	6 Test tubes (15-20 cm)	1China marker	

Reagents

Ammonium hydroxide	Distilled water
Methanol-water 50/50	Ethanol (denatured)
Methanol	Hydrochloric acid, 0.1M

Method

In order to chromatograph the inks on these documents you must get the ink back into solution.

1.) Cut a small portion from the end of a written word contained in the **original document** and place it in a test tube (10cm). Add **methanol** drop wise 2 or three drops may be sufficient. Place a stopper over the top. This will be designated the **scene ink** (test tube 1).

2.) Repeat step 1 above for the **second document.** The second document will be designated **possible forgery** (test tube 2).

** At this point, you should have two ink extracts: **scene ink,** and **possible forgery ink.** See figure 1 below.

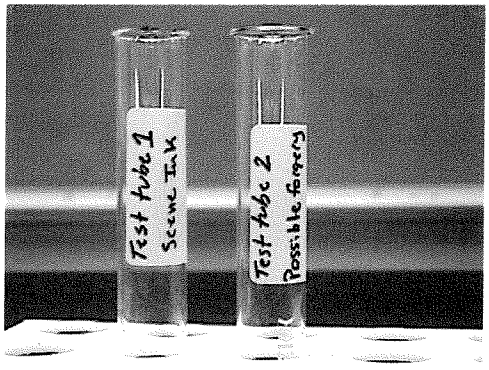

Figure 1, Ink Extractions

**Before proceeding have your teacher check your two ink extracts.

Teacher checkpoint: _____

3.) After your teacher has checked your two ink extracts store them at room temperature until needed.

4.) Obtain five test tubes between 15-20 cm long and a test tube rack. Using a china marker or sharpie label the test tubes as follows:

Solvents:

Test tube 1: Ammonium hydroxide
Test tube 2: Distilled water
Test tube 3: Water-methanol mixture
Test tube 4: Ethanol
Test tube 5: Hydrochloric acid

Place the test tubes in the test tube rack. Using a graduated cylinder or other appropriate liquid measuring instrument, measure 4ml of **each solvent listed above** and carefully pour it into the appropriate test tube. **At this point, you should have five test tubes with 4 ml of each solvent. See figure 2 below. Before proceeding have your teacher check your set-up. Teacher checkpoint: _____

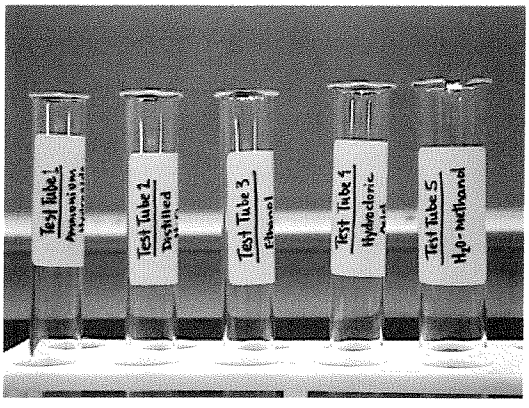

Figure 2, Solvents

5.) Obtain some filter paper and cut five strips making each strip 12.5cm long and 1cm wide (see figure 3). From the bottom of each strip measure up 1.5cm and using a pencil, make a light line across the filter paper (see figure 3). From the bottom of the filter paper up to the 1.5 mark cut a "V" in the filter paper, (see figure 3). From the bottom measure up 2.5cm and once again using a pencil, make a light line across the filter paper (see figure 3). From the bottom of the filter paper measure up 10.00 cm and using a pencil, make a light line across the filter paper (see figure 3). At this point, obtain 5 rubber stoppers with hooks in them. Suspend each strip of filter paper from the hooks (see figure 4). Each strip must hang suspended in the test tube without touching the walls of the test tubes. Minor adjustments may have to be made by adjusting the wire. **The goal is to keep the blotted ink extract 1cm above the solvent. At this point, you should have five filter paper strips attached to rubber stoppers with hooks. Also on each strip, attach a thumbtack on the bottom to keep the paper from rolling up. Have your teacher check your set up before continuing.

Teacher checkpoint: _____

DO NOT PLACE THE FILTER PAPER IN THE SOLVENTS AT THIS TIME.

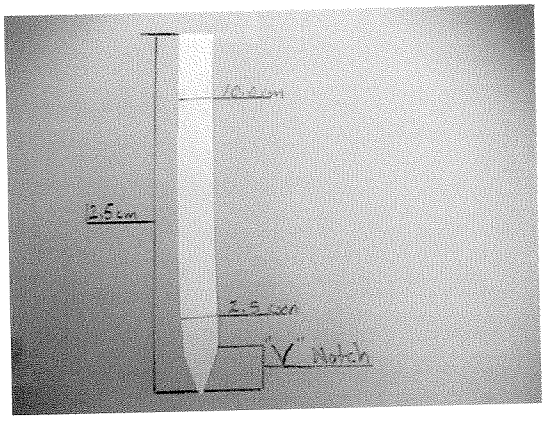

Figure 3

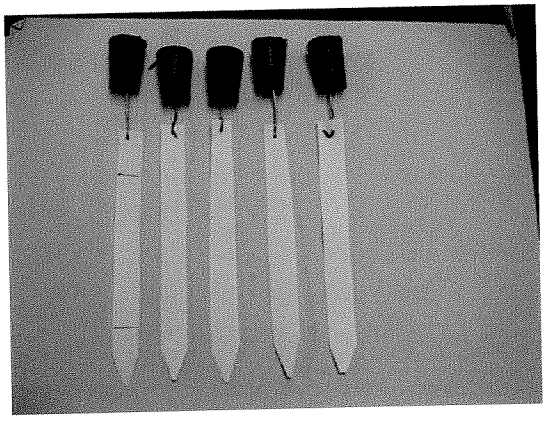

Figure 4

6.) Obtain the **scene ink extract from** step 1. Obtain a capillary tube and fill it by immersing one end of the capillary tube in the scene ink. At the 2.5cm mark, spot the filter paper by lightly touching the capillary tube to the filter paper. Spot and dry three or 4 times to build up the concentration of ink. ** At this point, you should have five strips of filter paper with a spot of scene ink on each one.

Teacher checkpoint: _____

7.) Lower the strips into the solvents, but do not immerse the spot. Let the solvent come over the spot by capillary action.

8.) Allow the solvent to rise up the strip until it reaches the 10cm mark or you have good separation.

9.) Remove the paper strip, mark the solvent front, and allow the strip to dry on a clean sheet of paper or suspend them from a support.

10.) Using the solvent that gave the **best** separation of the scene ink, chromatograph the ink extract from the possible **forgery documents.** Keep the strip of filter paper that gave the best separation of the scene ink for comparison to the possible forgery.

The best solvent to use for the possible forgery is: _____
** remember you are only using the best solvent on the possible forgery chromatogram, not all 5 solvents

10.)Attach both chromatograms to the front of the data sheet and hand them into your teacher.

11.)Clean all equipment and glassware. Dispose of all solvents in an appropriate waste container.

Data Section

Analysis of ink by paper chromatography

1.) Is this black ink really "black"?

2.) Is the order of colors on the strips the same in every case of solvent used?

3.) In your opinion, were the two documents written with the same ink? Why or Why not?

Chromatograms:

Scene ink **Possible forgery ink**

Key Terms for chapter 5

Chromatography	Compound	Electromagnetic spectrum	Electrophoresis	Element
Fluoresce	Frequency	Gas (vapor)	Infrared	Inorganic
Ion	laser	Liquid	Matter	Organic
Monochromatic light	Phase	Photon	Pyrolysis	Solid
Sublimation	Ultraviolet	Wavelength	X-ray	