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Electrophoretic Identification of Felt Tip Pen Inks

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ABSTRACT: Three techniques for the identification of felt tip pen inks by electrophoresis are described. Different solvents for extracting the ink to be tested are suggested. Ten different felt tip pen inks were used. While it was found more equipment, more chemicals, and a larger amount of the sample ink are necessary for electrophoresis than for some other processes of ink identification, electrophoresis is effective.

KEY WORDS: questioned documents, inks, electrophoresis

In the mid-1940s, when the ball-point pen was developed, document examiners soon realized certain new problems had been created. Of major concern was the analysis and comparison of ball-point pen inks.

In 1951, marker pens were introduced. These pens were the forerunners of today's felt tip pens. Felt tip pens use an ink-soaked wick, or a cartridge, from which the ink flows to a porous tip. The tip is often made of felt or plastic and is extremely flexible [*1*]. With the advent of felt tip pens, document examiners are again faced with the problem of ink analysis and comparison.

Much literature has been written describing procedures successful in the analysis of inks. The majority of this literature concerns the analysis by thin-layer chromatography (TLC) of liquid writing inks and ball-point pen inks. Although electrophoresis is mentioned occasionally as a method of ink analysis, a literature search divulged very little information on the subject. Harrison mentions electrophoresis as a means of ink analysis in his book, *Suspect Documents* [*2*]. In 1954, Brown and Kirk [*3*] mentioned in some detail ink analysis by electrophoresis. The most recent information found on analysis of ink by electrophoresis was by Brunelle [*4*] in 1972; he indicates electrophoresis has no advantages over TLC, but that electrophoresis can be used as another means to confirm the identification of inks. All of these authors dealt with liquid writing ink and ball-point pen ink. A search by the author failed to find any literature concerning felt tip pen ink analysis by electrophoresis. This is not to say there is no literature on the subject, but only that none was found.

This paper will concern itself with research done by the author in felt tip pen ink analysis by electrophoresis.

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Materials

The following equipment and chemicals were used in this project. Product names are not an endorsement by this laboratory or the author; they are used only as a means of identification for materials used.

Beckman Microzone electrophoresis cell, Model R-101
Beckman Duostat regulated DC power supply capable of delivering 0 to 500 V
Beckman cellulose acetate electrophoresis membranes
Gelman Super-Sepraphore® cellulose electrophoresis strips
Open-top plastic container measuring approximately 10 by 15 by 8 cm (10 by 15 by 8 in.)
Beckman electrophoresis membrane blotters
Helena sample applicator
Small vials (made from glass tubing)
Glycine
Sodium chloride
Barbital sodium
Diethyl barbituric acid (barbital)
Calcium lactate
Ethyl acetate
Ethanol (absolute)

Eight felt tip pen inks were used in this project. The pens all contained black inks.

1. Write Brothers
2. Schaeffer
3. Skilcraft
4. Pentel
5. Vega
6. Esterbrook
7. Parker
8. Sharpie

Extraction Procedure

Ink samples were prepared by marking on paper with each pen. Samples of each ink were put in individual glass vials to which a solvent was added. The solvent found most successful in extracting the ink from the paper (Solvent 1) was suggested by Brunnelle [*4*] and consisted of ethyl acetate/ethanol/distilled water in the proportions 75:35:30. Other solvents were also tried: Solvent 2 comprised butyl acetate/acetic acid/distilled water (4:1:4), Solvent 3 was composed of ammonia and distilled water (50:50), and Solvent 4 consisted of pyridine and distilled water (50:50).

It was noted that some inks extracted readily in some solvents and not at all in others. A very small portion of the sample to be analyzed should be tested prior to immersion to determine the solvent best suited for extraction of a given ink. It should be remembered that some felt-type pens, such as Sharpie, are used as permanent markers and the ink from these pens will not readily extract. Close observations should be made of the ink after immersion in the solvent. Although the pens used contained only black ink, most of the felt pen inks appeared as various shades of blue when extracted; however, some inks will extract as purple, amber, or brown, and this may be all that is necessary for verification or, more likely, for elimination.

Electrophoresis

Two buffer solutions were used for the electrophoretic separation. Buffer A, pH 6, consisted of 7.507 g of glycine, 5.844 g of sodium chloride, and 250 ml of distilled water, and Buffer B, pH 8.6, was composed of 2.19 g of barbital sodium, 0.345 g of diethyl barbituric acid (barbital), 0.096 g of calcium lactate, and 250 ml of distilled water.

The same electrophoretic procedure was used for both buffers. The electrophoresis cell was filled with buffer. The remaining buffer was poured into a plastic container (tank) to a depth of 1.6 to 3.2 mm (1/16 to 1/8 in.). A cellulose acetate membrane was placed in the tank and allowed to soak thoroughly in the buffer solution. Samples of each ink to be analyzed were placed in the individual cells of the sample applicator by using small pipets. The applicator was covered with a glass slide to prevent evaporation. The membrane was removed from the tank, the excess buffer blotted, and the sample placed on the membrane. The membrane was then fit onto the electrophoresis cell rack and the rack placed in electrophoresis cell. Care was taken to extend both ends of the membrane into the solution. The best separation occurred at 220 V for 15 min.

Both buffers gave good separation. Buffer A was perhaps a little more satisfactory than Buffer B inasmuch as the separation was less extended.

A third system was tried. The same buffers were used, but the cellulose acetate membranes were replaced by agarose gel plates. Although the plates can be purchased, those used in this procedure were made in this laboratory.

A stock buffer for the agarose plates can be made by preparing a solution of 7.0 g barbital sodium, 1.1 g diethyl barbituric acid barbital), 1.02 g calcium lactate, and 1000 ml distilled water. At the time of use, 10 ml of stock buffer is added to 0.2 g agarose. The mixture is brought to a boil and poured onto a gel plate. The gel solution should be evenly spread and free from bubbles or breaks, and the plate must be thoroughly cooled before the sample application.

In this procedure, small pieces of ink-bearing paper, measuring 1 by 3 mm, were removed from the document. Slots 3 mm in length were cut into the gel in a straight line across the width and near the center of the gel plate. The bits of paper bearing the ink to be analyzed were introduced edgewise, one in each slot cut in the gel plate. The plate was then put into the electrophoresis cell. It was necessary to make wicks extending from each end of the gel plate down into the solution. Filter paper or any highly absorbent material will fill this need. The best separation occurred in this test at 200 V for 8 min.

In each of the procedures, the processed plates should be examined under long wave and shortwave ultraviolet light for fluorescent patterns. It should also be examined through an infrared instrument to determine the transparency or opacity of the ink components. These characteristics are very important in the analysis of the ink sample and, like the color of the ink extract, may be all that is needed for identification.

The processing time for electrophoresis depends on the quantity or concentration of the ink sample, the buffer used, the amount of voltage applied, and the voltage application time. Usually, the higher the voltage applied, the less time needed for separation. There are minimum and maximum voltages that can be used for suitable separation. These parameters can be learned with a little practice.

In each of the procedures used in this project, it was necessary to alter the document by removing portions of the inked paper. As with any documents problem where the document under examination is to be altered, consultation with the submitter is mandatory. Although this is also necessary in TLC analysis, a great deal more inked material is needed in electrophoresis to get the ink concentration necessary for a good test. This is probably the greatest disadvantage in the electrophoretic process. This fact, along with the equipment needed and the time involved as compared with TLC, would indicate TLC to be a more practical process for felt tip pen ink analysis. The electrophoresis process is an effective system of felt pen ink analysis, however, and should certainly not be discounted.

References

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