TECHNICAL NOTE

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Technique for Transferring Ink from a Written Line to a Thin-Layer Chromatographic Sheet

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ABSTRACT: A method is presented for transferring ink directly from small samples of written lines that have been removed from questioned documents. The technique is flexible, uses limited samples if necessary, and is quite rapid.

KEYWORDS: questioned documents, inks, chromatographic analysis

The general techniques for examining ink specimens from written lines on questioned documents have been in existence for several years. A typical method would be that found in an article by Nakamura and Shimoda [*1*]. Their procedure involves the removal of several small plugs of paper containing the ink in question from the document using a sharpened hypodermic syringe needle. These plugs (also referred to as microplugs or microdots) are placed in the well of a spot plate and a small quantity of pyridine is added to dissolve the ink from the paper. After dissolution has taken place, a capillary pipette is used to transfer the pyridine-ink mixture to the appropriate thin-layer chromatographic sheet or plate for development.

An alternate method that is currently in use involves placing the microdots in a small glass vial and adding a few microlitres of pyridine to dissolve the ink. The resulting colored solution is then transferred to the thin-layer sheet or plate using a capillary pipette.

Although the above procedures have been in use for some time and produce acceptable results, they do have certain drawbacks. One of the drawbacks is that several microdots are usually required. There are occasions when the amount of writing and so forth available for analysis is very limited—sometimes as little as one digit or one letter. If several microdots are removed, little is left of the writing. This can reduce the value of the document for further evidential use.

The second deficiency of current methods involves the time that it takes to transfer several microlitres of solution from the well of the spot plate or the glass vial to the thin-layer sur-

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face. Each addition must be allowed to dry before more solution can be added. If this is not done, the spot will become overly large, and good, sharp development will not be possible.

Lastly, it is difficult to change extracting solvents. Although pyridine is an excellent solvent, I have found it desirable to be able to use other solvents or solvent systems on the same microdots.

With the foregoing in mind, I have developed a modified technique that overcomes some of the deficiencies of current, common practice. In the following I will describe the technique that I have been applying successfully for some time in my ink analysis work.

The removal of the microdots from the paper is unchanged and involves the use of a sharpened #18 syringe needle. I have found it very helpful to use a small piece of 6.35-mm (1/4-in.) thick plastic (methyl methacrylate or equivalent) to punch against. The plastic provides good, firm support but is soft enough to prevent damage to the punch. Depending on the intensity of the written line, I may only remove two or three microdots, and when the sample is limited, have made identifications with a single microdot.

The next step in the procedure is where the modified technique departs from the usual practice. A single microdot is picked up with a fine-tipped pair of tweezers and placed ink-side down directly on the thin-layer sheet or plate. In my practice I have been using silica gel sheets produced by E. Merck, Darmstadt, Germany (precoated TLC plastic sheets, silica gel 60, without fluorescent indicator, Catalog 5506).

With the microdot properly positioned, a small, 1-μL capillary pipette (Microcaps, Drummond Scientific Co., Broomall, PA) is filled with an appropriate solvent or mixture of solvents. The pipette is then placed directly in the center of the microdot and a portion of the solvent is allowed to flow through the microdot, dissolving the ink and depositing it on the silica gel. If it appears that the solvent system chosen has not removed all of the removable colorants from the microdot, another pipette can be filled with a different solvent and the second fluid allowed to flow through it. I routinely use two solvent systems in all analyses; first, a mixture of *n*-butanol, isopropanol, and water (2:1:1 by volume) followed by pyridine. If the ink removed from the single microdot does not appear to be sufficient for thin-layer analysis, a second microdot can be punched and placed in the same spot on the sheet and extracted.

The whole process is very rapid and allows the analyst to extract the maximum amount of color with a minimum amount of sample. Because of differences in papers, there are occasions when the flow-through process requires some time. Highly sized and coated papers definitely retard the rate at which solvents flow through them. However, I have yet to encounter a paper that is completely impervious.

After allowing the solvents to evaporate from the transferred spot, the sheet or plate is developed in the usual manner. The evaporative process can be hastened with the application of mild heat.

The developing chambers that I normally use are capped glass vials, 10.8 cm in height by 2.7 cm in diameter and were referred to in a previous publication [*2*]. Such chambers readily accommodate strips of thin-layer chromatographic sheet which are 10 cm long by 1.6 cm wide having been cut from full-size, 20- by 20-cm sheets. The eluent that I favor for general use is the same as the first extracting solution: 1-butanol, isopropanol, and water (2:1:1 by volume).

After development, the thin-layer strip is removed from the chamber and the eluent front is marked. When dry, the strip is compared to a reference catalog of ink separations for identification and possible dating. The integrity of the silica gel can be preserved by spraying the strip lightly with clear acrylic plastic.

To summarize, a thin-layer chromatographic technique has been presented which involves the following steps: (1) removal of a microdot sample from a written line on a questioned document, (2) placement of the microdot directly upon silica gel sheet or plate, (3) transfer-

ral of the ink from the microdot to the plate by directly allowing solvent to flow through the microdot, and (4) development in the usual manner.

This technique has the advantages of (1) requiring less sample, (2) using all of the colorant available, (3) allowing more than one extracting solvent to be used, and (4) speed.

References

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