TECHNICAL NOTE

Joel Harris¹

Developments in the Analysis of Writing Inks on Questioned Documents

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ABSTRACT: The development of a 1-mm-diameter micro-hole punch allowing for the rapid collection of paper plugs from documents bearing questioned inks is presented. The Drummond Digital Microdispenser is also described, highlighting its advantages over other extracting and spotting techniques used in forensic ink analysis.

KEYWORDS: questioned documents, inks, punches, extraction

In the past two years, the Central Forensic Laboratory of the Royal Canadian Mounted Police has taken steps to develop an ink library to assist in the dating of questioned documents. Towards this end, some of the reported procedures involved with the sampling and extraction of ink from paper and the spotting of the ink on thin-layer chromatography (TLC) plates [1-5] have been modified. The following discussion describes these modifications and how they have significantly reduced the time required to complete a typical ink analysis.

Micro-Hole Punch

The sampling of ink from a questioned document involves, at times, a procedure which is tedious by the very nature of the operation and is further complicated by the tool chosen to perform the task. A single ink analysis typically involves the removal of an eight-plug fraction for each ink in question on a document [2]. In some instances, this may require the removal of hundreds of plugs (that is, ledgers, notebooks, and so forth).

Paper plugs bearing ink are generally removed from a document using a 20-gauge syringe [1-5] with the pointed end filed flat (Fig. 1). The ground end of the syringe is placed on top of the ink stroke with the questioned document resting on a piece of glass or hard paperboard (Fig. 2). The syringe is gently rotated with firm downward pressure removing a plug of paper bearing ink. The end of the syringe containing the cored-out

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¹Questioned document examiner, Central Forensic Laboratory, Royal Canadian Mounted Police, 1200 Vanier Pkwy., Ottawa, Ontario, Canada, K1G 3M8.

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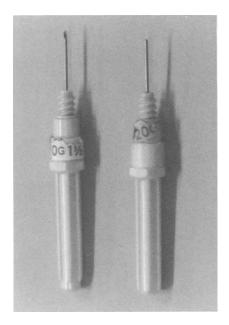


FIG. 1—Example of a 20-gauge syringe (left) with the pointed end filed flat (right).



FIG. 2—The ground end of the syringe is placed on top of the ink stroke and gently rotated with firm downward pressure removing a plug paper bearing ink.

paper plug is placed in a 1-dram vial and a wire plunger from a glass injection syringe is placed through the top end of the syringe and used to push the recessed plug into the vial (Fig. 3). This coring procedure must be performed in a slow, steady manner so as not to tear the document.

To reduce the time required to collect the micro-plugs, the purchase of a micro-hole punch of similar diameter was considered; however, no existing manufactured product could be found. The decision was then made to design and produce a 1-mm-diameter micro-hole punch with the following specifications in mind:

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• capable of punching out sharply defined 1-mm-diameter plugs of paper;

• able to collect each punched out plug directly into a 1-dram vial seated below the punch;

• accessible to routine cleaning and comprised of materials that would not be affected by suitable cleaning solvents; and

• capable of obtaining samples anywhere on a 8.5-by 14-in. (21.5- by 35.5-cm) document.

A punch with these characteristics was manufactured by the Security Equipment Branch of the Royal Canadian Mounted Police (Fig. 4). The punch is activated with a quick downward tap of the punch head. A spring returns the punch head to the rest position so that the sampling operation can be repeated. The punch is best operated from an elevated position which allows easy viewing of the area of interest (Fig. 5). The plugs produced have sharp edges and the holes left in the document are uniform and clean. A 1-dram vial is screwed into a vial cap recessed below the punch port (Fig. 6) to receive the punched-out plugs of paper.



FIG. 3—The cored-out paper plug is transferred to a 1-dram vial and pushed through the tip of the syringe using a wire plunger.

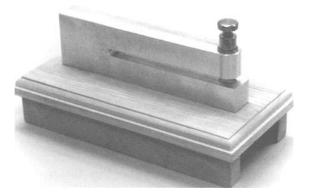


FIG. 4-The micro-hole punch.

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FIG. 5—The punch is best operated from an elevated position which allows easy viewing of the sample area.

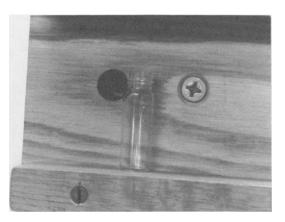


FIG. 6—A 1-dram vial screws into a vial cap seated below the punch to receive the paper plugs.

This punch met all of the required specifications. A paper plug can be removed from any point on an 8.5- by 14-in. (21.5- by 35.5-cm) document. Several 8-plug samples of different inks can be collected directly into the extracting vials in a matter of minutes which significantly reduces the sample preparation time by the syringe method.

Drummond Digital Microdispenser

The next stage in an ink analysis involves extracting ink from paper plugs and spotting the ink on TLC plates. The ink from the paper plugs is then washed with 2 to 5 uL of the appropriate extracting solvent. Traditionally, the extracting solvent is collected in calibrated 10-uL glass pipettes from a beaker reservoir by capillary action or with the aid of a siphon tube system [2] (Fig. 7). In many instances, the siphon tube system is dispensed with only to be reused once the analyst has experienced the intoxicating effects of pyridine. This extraction procedure is cumbersome, time-consuming, and requires considerable patience to be used effectively and precisely (Fig. 8).

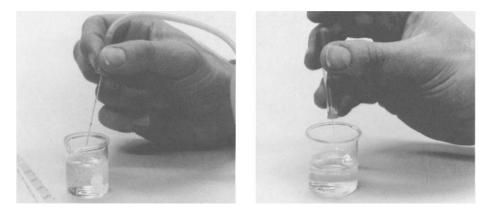


FIG. 7—Extracting solvent is collected in 10-uL glass pipettes from a beaker reservoir using a rubber nipple and capillary action (left) or with a siphon tube system (right).



FIG. 8—It is tedious, time-consuming, and difficult to extract efficiently the ink from paper plugs using a glass pipette equipped with either a rubber nipple or a siphon tube system.

The spotting of the extract on the plates using glass micropipettes is not as difficult but still requires a steady hand and careful manipulation (Fig. 9). An Eppendorf pipetter (Fig. 10) was tested as a suitable alternative to the handheld glass pipettes; however, loading and spot size were difficult to control (Fig. 11). This type of pipetter is a twostop system requiring that the plunger be depressed in two successive steps to expulse the extract. During the second step of this operation, the extract is deposited quickly, producing large, diffuse spots on the plate. A member of our Toxicology Section suggested a digital microdispenser be used to control the quantity of extract applied to the TLC plate.

The Drummond[®] Digital Microdispenser (Fig. 12) was chosen as a possible alternative. This tool combines the use of glass pipettes with an accurate one-step digital dispensing gun. The volume of extracting solvent required can be adjusted in 0.01-uL increments up to 10 uL. This instrument is especially useful when the same volume of extracting solvent must be used to analyze several ink samples. This one-stop dispensing system allows the extract to be added to the TLC plate in small increments which permits better control of the spot size and reduces loading since the extracting solvent can be blown

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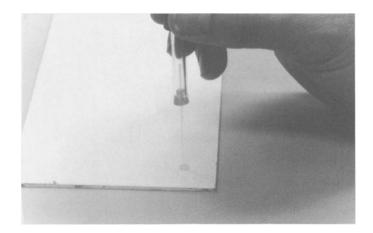


FIG. 9-Spotting extract on TLC plates using micro-pipettes and a rubber nipple.



FIG. 10—The Eppendorf pipetter.

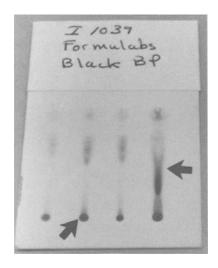


FIG. 11—The deposition of extract onto a TLC plate from an Eppendorf pipetter is difficult to control and can result in spots that are large, diffuse, and generally overloaded. This results in poorly separated dye components with associated tailings (arrow).

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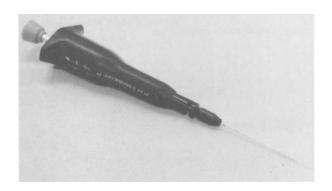


FIG. 12-The Drummond Digital Microdispenser.

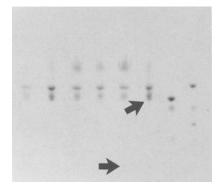


FIG. 13—Spotting TLC plates using a Drummond Digital Microdispenser produces small, sharply defined spots, which are well separated dye components on the TLC plate.

off after each addition to the plate (Fig. 13). The Drummond Microdispenser is operated easily with one hand thus offering rapid and efficient extraction of the ink from the paper with minimal loss of solvent to the paper substrate. Because the extraction is more efficient, less sample is required and fewer plugs need to be removed from the questioned document.

The use of the micro-hole punch and the Drummond Digital Microdispenser combine to improve the precision and accuracy of convential methods and significantly reduce the time required to analyze writing inks by thin-layer chromatography.

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Address requests for reprints or additional information to Joel Harris Central Forensic Laboratory Royal Canadian Mounted Police 1200 Vanier Pkwy. Ottawa, Ontario, Canada, K1G 3M8