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Technique for the Separation of Ink Dyestuffs with Similar R_F Values

The use of Eastman Kodak Chromagram®[2] sheet for the separation of the various dyestuffs used in the compounding of inks has already been documented [*1*]. It has developed into one of the more useful techniques for the identification of inks. Both silica gel and cellulose Chromagram® sheets are now commonly used with a variety of eluent systems. Notwithstanding the variety of techniques available to the chromatographer, there are still certain mixtures of dyestuffs which do not yield clear-cut separations. The technique which will be reported here allows better separations to be made when used on these difficult mixtures.

To date, the technique has been applied to separations of water-base (fountain and soft-tip pen) and ball-point pen inks with equal success. Both silica gel and cellulose Chromagram® sheets have been used. The specific procedure involves the following materials and techniques.

For use, the Chromagram® sheet is cut into small strips 10 cm long and approximately 1.6 cm wide. This size is dictated by the developing chambers used which are capped glass vials, 10.8 cm in height and 2.7 cm in diameter.

Approximately 2 cm^3 of an appropriate eluent are placed in each vial. Commonly used is a 2:1:1 (by volume) mixture of *n*-butanol, isopropanol, and water. The ink is spotted about 1 cm from the end of the strip and the ink solvent is allowed to evaporate. About one third of a microlitre is used. Ballpoint inks, due to their high viscosity and color intensity, are diluted with 10 to 15 parts of ethyl alcohol or other suitable organic solvent. With suitable modifications in technique, extracts from written lines can also be used.

The usual procedure would be to place the spotted strip in the glass vial and to remove it after the eluent had risen up the Chromagram® strip to an appropriate height. The problem with this procedure arises when the dyestuffs involved have similar R_F values under the conditions of the test and thus are not completely separated. For example, in a green ink blue and yellow spots may be detectable, but if they do not completely separate they will overlap to produce a greenish area. The chromatographer cannot be sure that a green dye does not also exist in this area of overlap. Diagrammatically, the situation would be as shown in Fig. 1.

To overcome this problem, the following modification to standard technique has been developed. Instead of placing the spotted strip into the developing chamber after drying, certain areas of the silica gel or cellulose layers are carefully removed from the

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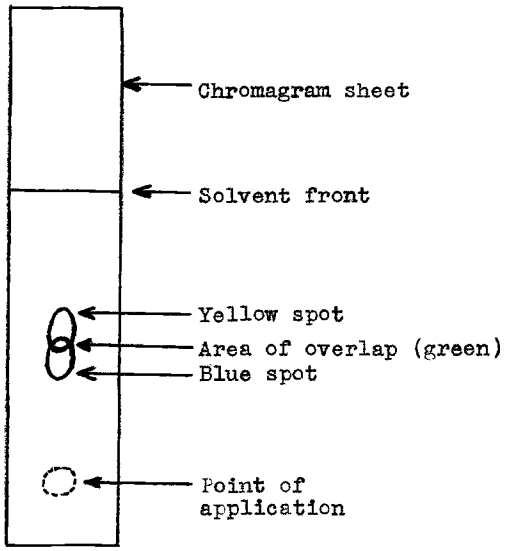

FIG. 1—*Separation using standard procedure.*

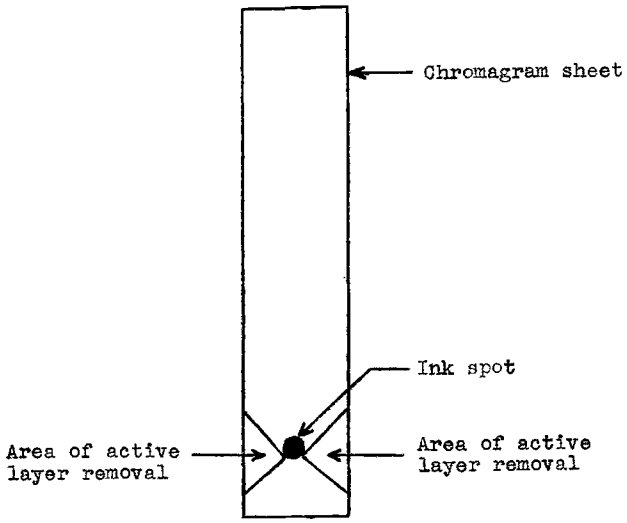

FIG. 2—*Pattern for active layer removal.*

underlying support sheet. The removal is usually done with a scalpel or other sharp instrument. A glass microscope slide may be used as a straightedge to guide the scalpel. The pattern of active layer removal is shown in Fig. 2. This pattern was developed after considerable experimentation with a variety of configurations.

After the active layer has been removed as shown, the strip is placed in the developing chamber and run in the usual fashion. The removal of the active layer in the pattern shown serves to channel all of the eluent through the spot and produces a far different

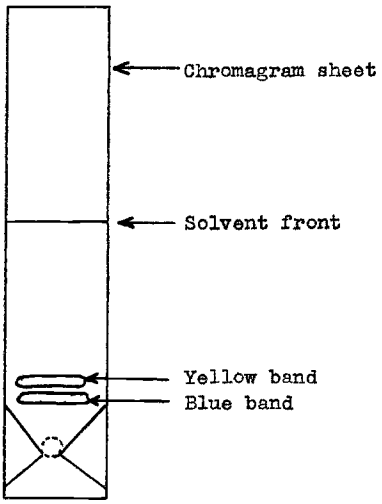

FIG. 3—*Separation using modified procedure.*

result than the standard technique. Thus, the same green ink used as the example for Fig. 1 would separate, as shown in Fig. 3. As can be seen, instead of elliptical overlapping spots, a series of discrete bands is generated. In addition to improved separations, it has also been observed that the technique is faster since the solvent front need not be allowed to advance as far as in the standard technique.

In summary, what is reported here is an improved technique for the chromatographic separation of ink dyestuffs, especially those which are difficult to separate using ordinary methods. An added positive feature of the method is that it is faster than the standard procedure.

Reference

[*1*] Tholl, J. "The Eastman Chromagram Sheet for Chromatography: Review and Experimentation," *Police*, Vol. 10, 1966, pp. 6-11.

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