Richard L. Brunelle,[1] M.S. and Huy Lee[2]

Determining the Relative Age of Ballpoint Ink Using a Single-Solvent Extraction, Mass-Independent Approach

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ABSTRACT: Brunelle et al. (*Journal of Forensic Sciences.* Nov. 1987) recently reported a single-solvent extraction technique for determining the relative age of ballpoint ink entries on questioned documents. This technique was mass dependent, which means equal amounts of ink had to be removed from the document for all samples compared. This paper describes a modification of the previous procedure that makes the age determination independent of the amount of ink sampled for testing. The modified procedure involves extracting the inks with solvents, spotting the ink extract onto a thin-layer chromatographic plate, and then separating the dye components in a solvent system of ethyl acetate:alcohol:water (70:35:30 parts. respectively). The relative concentrations of the dye components are measured using a densitometer. The calculated ratios of the relative concentrations are independent of the amount of ink taken for analysis. Aging curves were prepared for four different ballpoint inks and two non-ballpoint inks. The effect of different papers on ink aging was determined, and the mass independence theory was verified. The feasibility for determining the relative age of non-ballpoint inks was also established.

KEYWORDS: questioned documents, inks, extraction, mass independent, relative age, ratios, solvent extraction, densitometry, thin-layer chromatography. paper, ballpoint and non-ballpoint ink

As new and better techniques are developed for determining the age of inks on questioned documents, more forensic scientists are using these techniques. Most of the improvements in this field over the past eight years deal with determinations of relative age as opposed to absolute age or first production dates of specific ink formulations. All of these developments have been reported [*1-7*] and will not be discussed further in this paper. A critical evaluation of all techniques used for dating ballpoint inks up to 1987 has also been reported [*8*].

Although a variety of relative aging techniques have been tried and reported by several researchers, none of the approaches to date has achieved wide acceptance. This is because considerable experience is required to get reproducible results and because no standard procedure has been developed that applies to most, if not all, types of writing inks.

This paper describes a simple solvent extraction procedure for determining the relative

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[1]Owner, Brunelle Forensic Laboratories, Fairfax, VA.
[2]Student, Criminal Justice Program, Michigan State University, East Lansing, MI.

age of ballpoint inks. The procedure is reproducible and independent of the amount of ink taken for analysis. The effect of paper and solvent selection on the determination of the relative age of ink is also described.

Experimental Procedure

A combination of oxidation, cross-linking, polymerization, and solvent evaporation takes place as ink ages on paper. These factors cause the components in the ink to become less soluble in organic solvents as the ink ages. This is one phenomenon that permits the age of most ballpoint inks to be estimated. Two conditions must be satisfied before the technique can be applied in actual case investigations: the inks compared must consist of the same formulation and be on the same paper so that the inks are continuously exposed to the same storage conditions; and among the inks present, there must be some with known dates of placement on the document for comparison.

For some inks, the subtle fading or degradation of the dyes is a factor of aging. Therefore, solubility is not the only factor for these inks in the estimation of age. This change in the dyes is usually not visible to the naked eye and therefore does not prevent a determination that the inks being compared are the same formulation. The densitometer is sensitive enough to detect these subtle changes, however. When fading or decomposition of dyes are measured, pyridine should be used as the extracting solvent for ballpoint inks and water-resistant non-ballpoint inks. Water-soluble non-ballpoint inks should be extracted with a 1:1 mixture of ethanol and water.

It is recommended that aging curves using pyridine as the extracting solvent be tried first to determine if dye fading or decomposition produces ratios that change with the age of ink. If this approach fails to show a relationship with age of the ink, then aging curves based on solubility of the ink should be obtained using weaker solvents such as *n*-butanol, *n*-propanol, toluene, or other suitable solvent.

Ink Sampling

Take a minimum of either approximately 1-cm length of ballpoint ink line or approximately 10 plugs of ink (1-mm diameter), removed with a hollow syringe needle (microplugs). Up to 20 microplugs of ink should be taken, if sufficient quantity of ink is available for analysis. This will improve the accuracy of the densitometer readings.

Place the ink samples into 0.5-dram glass vials. If a 1-cm ink line is used, cut the line in a consistent way into smaller pieces which will fit on the bottom of the vial. It is advisable to use a consistent sampling method to avoid the effects of working with different surface areas.

Extraction Procedure

Add 20 μL of a suitable weak solvent to the ink sample in the vial (a Centaur automatic pipet was used in this study). To find the correct solvent, place 1 μL of the candidate solvent on a written line to see how much of the ink dissolves. When solubility is the measure of age, use a solvent that will only slightly dissolve the ink line. *N*-Butanol appears to be a useful solvent for some ballpoint inks. If fading or dye deterioration is the aging factor to be determined, then a strong solvent such as pyridine should be used to totally extract the ink.

Cap the vials to prevent evaporation and allow the ink to extract for 30 min. Rotate the solvent in the vials every 10 min to ensure consistent extraction. When total extraction of ink is desired using strong solvents, an ultrasonic bath may be helpful.[3]

[3]This suggested procedure was used in Fig. 11 for both extractions.

Thin-Layer Chromatography

Remove at least two (three, if possible) 5-μL aliquots of the extracted ink and spot onto a clean silica gel thin-layer chromatographic (TLC) plate, without fluorescent indicator. (Plates such as E. Merck or Whatman that have uniform silica gel coatings are satisfactory.) The TLC plate should be preconditioned by placing it into an 80°C oven for 15 min and then allowing the conditioned plate to cool to room temperature in a desiccator. In this study, Drummond Microcapillary pipets were used to spot the inks on the TLC plate. It is important to spot the ink extract in one smooth deposition on the TLC plate to get the same uniform size spots.

Repeat the procedure described above with all inks compared. Spot all samples in direct line on the same TLC plate using graphite pencil marks as a guide.

Dry the ink spots for 5 min in an 80°C oven. Allow the plate to cool about 5 min in a desiccator. Place the cooled plate into a TLC developing chamber containing a solvent system of ethyl acetate:ethanol:water (70:35:30 parts, respectively). The solvent system should be allowed to equilibrate for at least 30 min, and the solvent system should be replaced after developing no more than three chromatograms. (Note: It may be desirable to take densitometer readings of the unseparated ink spots before placing the plate into the development tank. This will permit an age curve to be prepared on the composite ink spot. This procedure will produce a reliable aging curve, if nearly equal samples of ink were removed for testing [*1*].)

Allow the TLC plate to develop until a minimally distinct separation of the dye components is observed. If the plate is left too long in the solvent system, the dyes will be better separated, but they may be too diffuse for accurate measurement with the densitometer (see Results and Discussion). The appropriate development time will vary with each ink formulation, so careful observation is needed in this step.

Densitometry

After TLC development, remove the plate from the developing tank and allow to air dry in a fume hood for about 5 min or until the solvent is no longer visible on the plate. (Avoid prolonged exposure of the separated dyes on the plate to light and heat. Some dyes fade quickly once separated from the other ink ingredients.)

If plastic TLC plates are used, tape the dried, developed TLC plate onto a (20- by 20-cm) glass plate backing. (This glass backing provides the support necessary to prevent buckling of the plastic plate in the densitometer.)

Place the supported TLC plate into a densitometer and scan the dyes for each ink sample using a Shimadzu Model CS-930 or equivalent. Scanning should be done in the linear scan and the absorption/reflection mode using a slit width that approximates the width of the dye spots. The width of the slit should be the smallest available setting on the densitometer to provide optimum resolution (approximately 1.0 mm). Take the densitometer readings (at 585 nm for blue and purple dyes and 500 nm for red and orange).[4]

Calculate all possible ratios of the dyes present in each sample (Dye 1/Dye, 2, Dye 2/Dye 3, Dye 1/total area, and so forth). Choose the dye components that provide reproducible ratios and ratios that show the most change with the age of the ink for a given TLC developing time at any concentration. The ratios can be determined by dividing the area of one dye component by the area of another dye component or by calculating the area of a particular dye as a percentage of the total area. Plot the ratios determined for each known dated ink sample versus age (months):

$$\text{Ratio} = \frac{\text{Area of Dye Component}}{\text{Total Area of All Dyes}} \text{ or } \frac{\text{Area of Dye } X}{\text{Area of Dye } Y}$$

[4]In this study, the Bic Banana's red and orange dyes were scanned with 560-nm light.

The age of the questioned ink (*Q*) sample is estimated by referring to the graph plotted for the known dated inks. If the age of the *Q* ink is within the range of the known dated inks on the graph, the age can be estimated with an accuracy that depends on the slope of the curve at the point where the *Q* ratio intersects the curve (see Fig. 5).

Results and Discussion

The purpose of this study was to modify the previously reported single-solvent extraction technique to produce a relative aging procedure that would be independent of the amount of ballpoint ink analyzed. A second purpose was to determine the reproducibility of the technique and the effect of different types of paper on the ink aging process. Lastly, we wanted to determine if the relative aging technique could be applied to non-ballpoint inks. The following discussion addresses these issues and elaborates on ways to optimize aging curves to produce the most accurate ink age estimates.

Reproducibility

Accurate, reproducible, and reliable measurements are critical in any analytical procedure. Therefore, this study began with several experiments to determine the reproducibility of the densitometer readings and relative aging curves which reflect the accuracy of the entire procedure. Tables 1 and 2 and Figs. 1 and 2 show the reproducibility of the densitometer readings, when the beam slit was parallel and perpendicular to the inks spotted on the TLC plate and using varying concentrations of ink. Relative standard deviations averaged 0.002 using the parallel beam slit and the perpendicular beam slit. Therefore, there is no advantage to using one type of beam slit over the other. The measurements were proportional to the concentration of the ink examined in the range of 10 to 25 microplugs of ink. This demonstrates that, in this case, every microplug contained equal or nearly equal amounts of ink.

The TLC plate used to obtain Tables 1 and 2 and Figs. 1 and 2 was developed to separate the dyes; its densitometer measurements are summarized in Table 3 and Fig. 3. Table 3

TABLE 1—*Reproducibility of densitometer scanning parallel beam slit.*

Number of Microplugs	Average Area (4 Scans)	Standard Deviation	Relative Standard Deviation
10	7 530.92	13.11	0.0017
15	10 824.30	18.13	0.0017
20	13 094.49	20.44	0.0016
25	17 247.58	12.78	0.0007

TABLE 2—*Reproducibility of densitometer scanning perpendicular beam slit.*

Number of Microplugs	Average Area (3 Scans)	Standard Deviation	Relative Standard Deviation
10	7 291.60	15.65	0.002
15	10 069.81	19.69	0.002
20	11 975.57	28.68	0.002
25	16 066.17	32.44	0.002

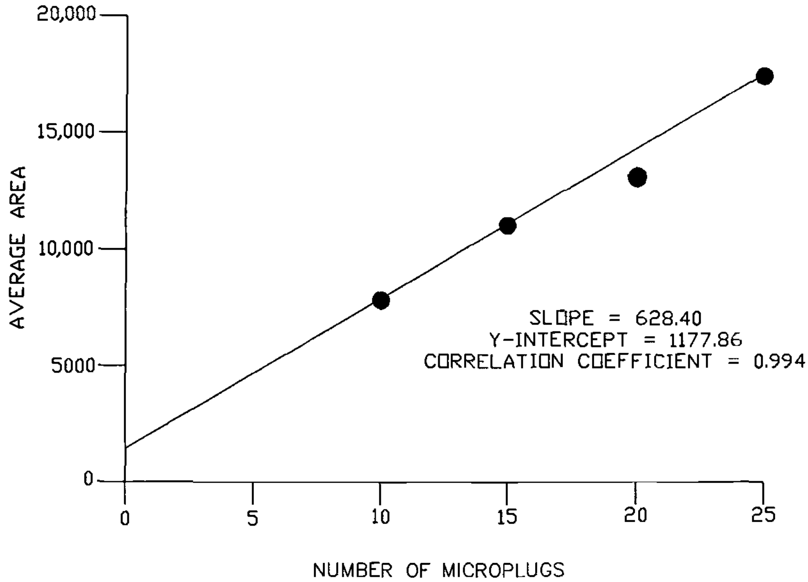

FIG. 1—*Average area (four scans) versus number of microplugs (parallel beam split).*

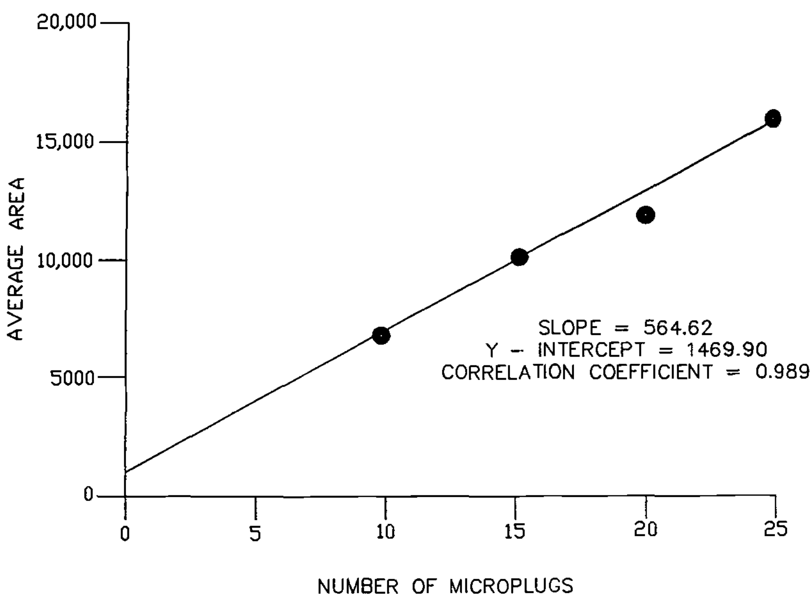

FIG. 2—*Average area (three scans) versus number of microplugs (perpendicular beam split).*

TABLE 3—*Mass independence by dye area ratioing.*[a]

Number of Microplugs	(A_1/A_2)	(A_1/A_3)	(A_2/A_3)
10	.996	1.050	1.054
15	1.096	1.043	0.952
20	1.046	1.010	0.961
25	1.047	0.988	0.943
S.D.	0.0258	0.024	0.038
Relative S.D.	0.025	0.023	0.039
	(A_1/A_T)	(A_2/A_T)	(A_3/A_T)
10	0.338	0.340	0.340
15	0.348	0.318	0.334
20	0.339	0.324	0.337
25	0.337	0.322	0.341
S.D.	0.012	0.022	0.022
Relative S.D.	0.035	0.067	0.065

[a]A_1 = Violet dye R_f 0.65 from Table 5, A_2 = violet dye R_f 0.70 from Table 5, and A_3 = violet dye R_f 0.73 from Table 5.

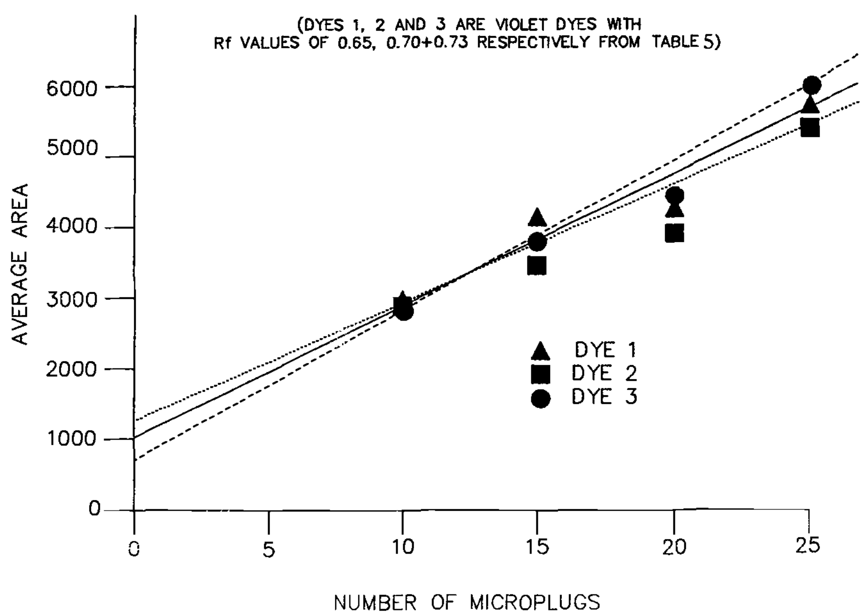

FIG. 3—*Average area (five scans) of dye components versus number of microplugs.*

shows the excellent reproducibility of the various dye ratios for varying concentrations of ink (the average of the relative standard deviations is 0.04, or 4%). Since these ratios do not change significantly with concentration, the procedure is mass dependent, indicating that Beer's Law is being followed.

Table 4 and Figure 4 show a *Beer's Law-like relationship* and thus supports that, in this case, the microplugs have equal, or nearly equal, amounts of ink. It also shows that the working range is 5 to 25 microplugs. Above 25 microplugs, all reflectance begins to look alike.

All of these experiments involving Tables 1 to 4 and Figs. 1 to 4 were done using fresh Pilot black ballpoint ink on Government Bond paper. The ink was extracted with pyridine, and only the three major violet dyes were used for the dye ratio calculations.

The instrument reproducibility (Tables 1 and 2) and the inter-sample reproducibility (Table 3) are both excellent. This excellent reproducibility is assumed in the experiments that

TABLE 4—*Adherence to Beer's Law.*

Number of Microplugs	Average Area	Standard Deviation	Relative Standard Deviation
3	3 206.33	482.17	0.150
5	5 578.86	234.32	0.042
7	6 010.25	62.48	0.010
10	10 358.14	41.27	0.004
15	14 045.95	134.92	0.009
20	15 991.85	80.31	0.005
25	20 303.21	167.76	0.008
30	23 330.40	55.93	0.002
35	24 158.06	223.90	0.009

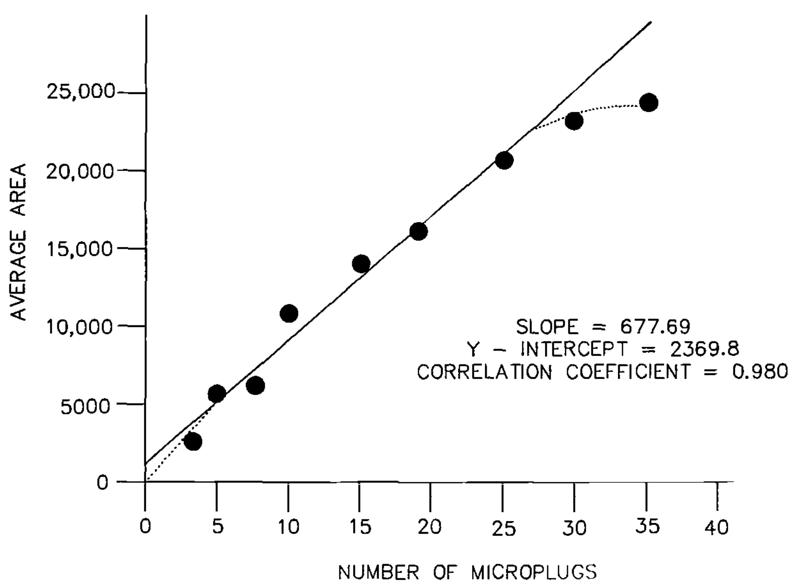

FIG. 4—*Adherence to Beer's Law: average area (four scans) versus number of microplugs.*

follow that involve different inks, paper, and solvents in that no reproducibility testing in these experiments was done. In actual casework, testing reproducibility is recommended, whenever possible.

Aging Curves

Figures 5 and 6 represent aging curves of a Pilot black ballpoint ink and a Bic blue ballpoint ink, respectively, using the described mass-independent procedure. The Bic curve shows significant aging taking place over a period of two years, whereas the Pilot ink showed no significant change in dye ratios after about one year. Under these circumstances, the age of questioned inks of the same formula and on the same paper could be accurately estimated up to two years for the Bic ink and one year for the Pilot ink.

Applications To Non-Ballpoint Inks

To determine whether this method could be used to estimate the age of non-ballpoint inks, two different non-ballpoint inks were examined (see Figs. 7 and 8). Figure 7 shows that an aging curve with substantial change in dye ratios with time was obtained for a Sanford (Sharpie) non-ballpoint ink. For this ink significant and measurable aging occurred over a period of four years. However, when the same procedure was applied to a Bic (Banana) non-ballpoint ink, a curve resulted that changed directions when the ink reached the age of two years. It is speculated that the change in slope of the curve was caused by calculating ratios of dyes that had vastly different rates of aging. The actual direction of the curve can be determined by testing age-accelerated samples or simply allowing the ink to age on the document.

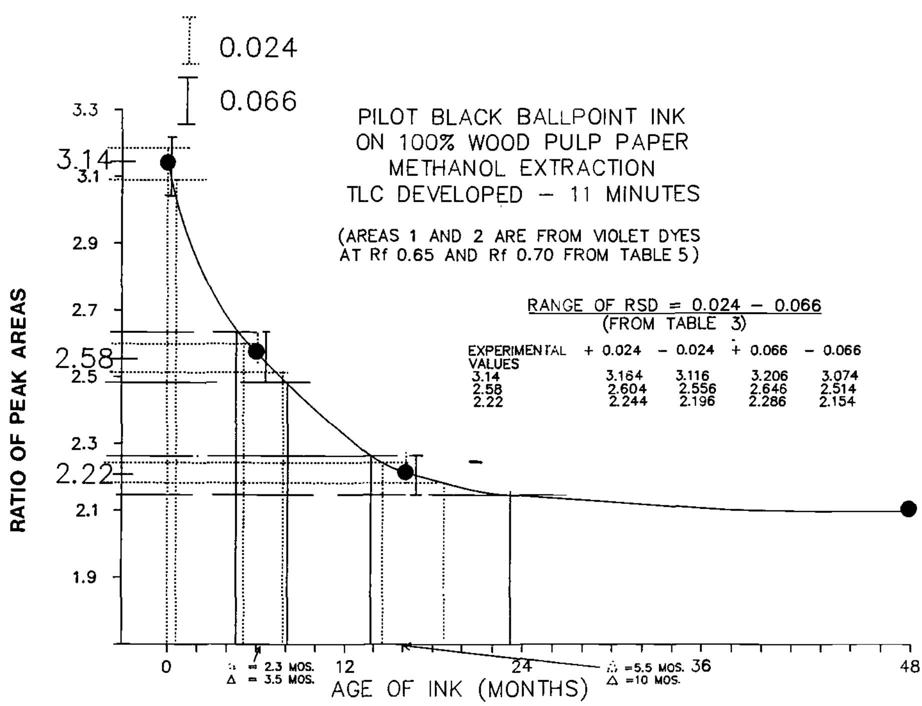

FIG. 5—*Aging curve.*

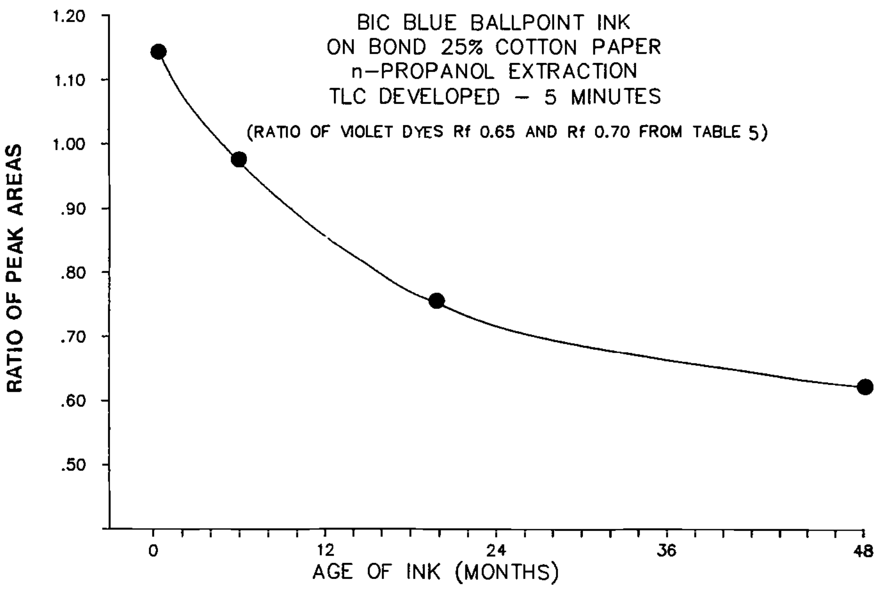

FIG. 6—*Aging curve using reciprocal of area ratio values.*

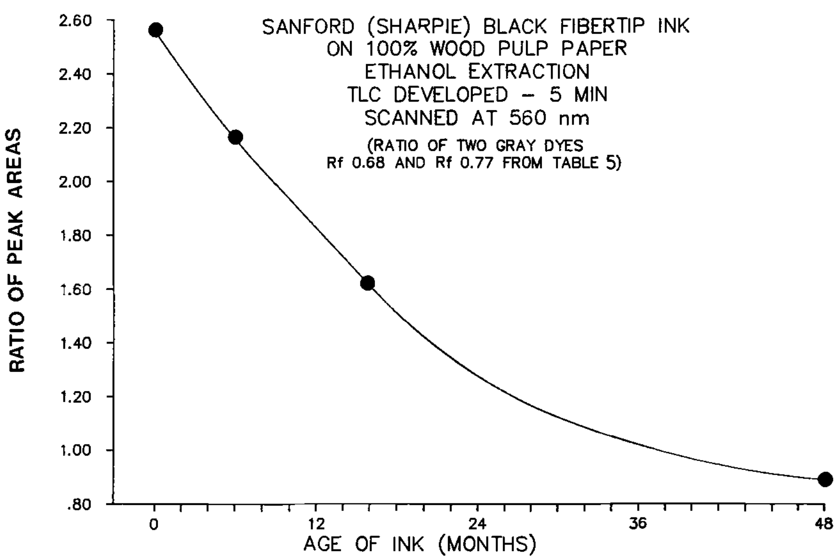

FIG. 7—*Aging curve for non-ballpoint ink.*

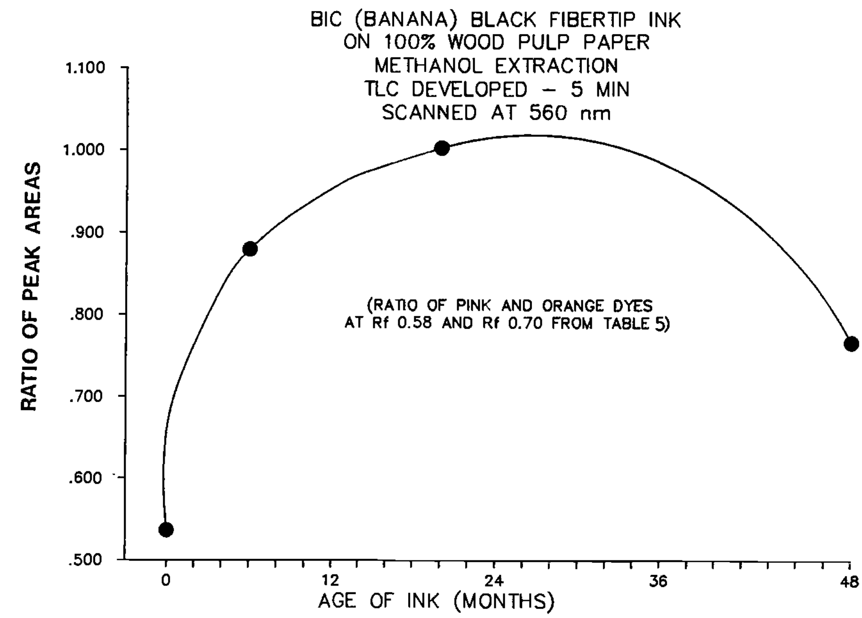

FIG. 8—*Aging curve for non-ballpoint ink.*

Effect of Paper on Aging Curves

Figure 9 shows the effect of different types of paper on the ink aging curves. Bic blue ballpoint ink was written on three types of paper and allowed to age for varying amounts of time up to four years. Dye concentration ratios were calculated and plotted versus ink age. The results show that ink on 25% cotton bond paper aged at about the same rate as the same ink on Whatman filter paper or ordinary 100% wood pulp paper in that no significant aging occurred after two years. However, the slope of the wood pulp paper curve was much steeper which would allow more accurate estimates of questioned inks during the first two years.

Figures 6 and 9 (Bic blue ballpoint ink on bond paper), which were done at different times, show reproducible curves upon adjusting the ordinate (y) scale. This supports the assumption made above that the instrument and intersample reproducibility determined hold for other inks, paper, and solvents.

It appears that the slope of the aging curves is affected by the different binders and fillers in the paper which change the relative solubilities of the dyes present in the ink. This finding makes it essential that only inks on the same type of paper should be compared for relative age.

Effect of Different Extraction Solvents

The effect of using different extraction solvents for producing aging curves is shown in Fig. 10. These curves reflect calculated dye concentration ratios of Formulab Black 587 ballpoint ink ranging in age from 0 to 4 years. The slope of the aging curves was somewhat different for each solvent until the ink reaches 15 months of age. Then the slopes become about the same for each solvent. Here the best curve (the one with the largest spread) was produced using butanol as the extraction solvent.

Methanol is a fairly strong solvent for extracting ballpoint inks, yet it still produced a good aging curve in Fig. 10. It was this finding that led to the speculation that perhaps subtle dye

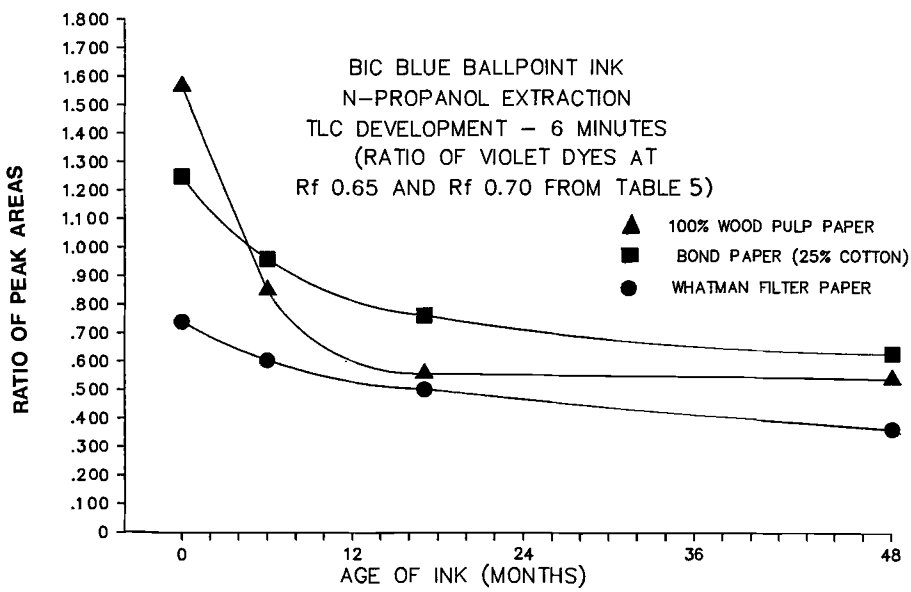

FIG. 9—*Effect of paper on aging curves.*

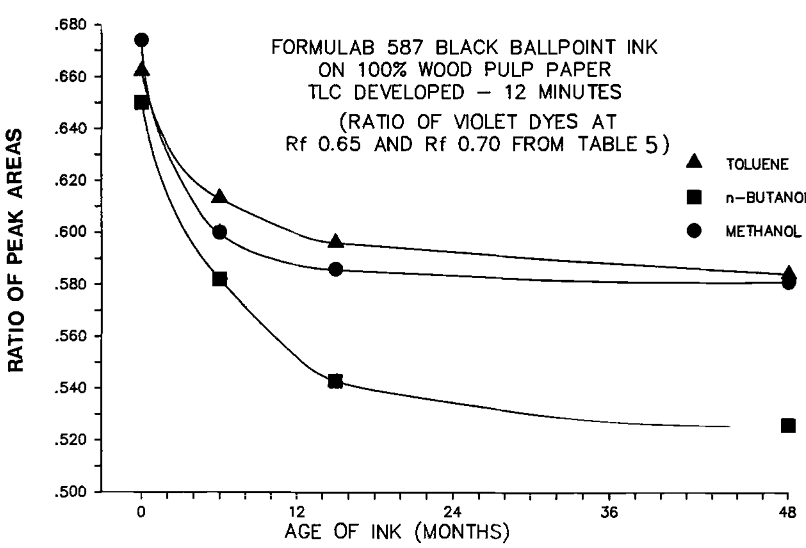

FIG. 10—*Aging curves using different extracting solvents.*

deterioration or fading or both may also be a factor of aging. Because certain dyes fade or decompose faster than others, the dye ratios change with age. To verify this speculation the experiment was repeated using pyridine, an even stronger solvent, on a Formulabs 587 black ballpoint ink (Fig. 11) This curve was also compared with a curve produced using *n*-butanol, a weak solvent compared to pyridine. An excellent aging curve resulted from both the pyridine and *n*-butanol extracts with measurable aging taking place over a period of five years.

Therefore, the authors believe that extractability and subtle fading/decomposition of dyes are both aging factors. It is emphasized, however, the fading or decomposition of dyes is very subtle and does not interfere with matching two or more inks as the same formulation.

Various Dye Ratios

Figure 12 shows aging curves for a Bic black ballpoint ink. Three different curves were plotted which represent different ways of calculating the dye concentration ratios. The densitometer measures areas for each dye component and the total area of all dyes present. Therefore several different ratios can be calculated. This ink contained three major dye components and the dye ratios can be calculated as Dye 1/Dye 2, Dye 1/Dye 3, Dye 2/Dye 3, Dye 1/total dyes, and so forth. Different dyes could age at different rates, so it is necessary to calculate and plot all possible dye ratios to determine which dye ratios produces the best aging curve. Figure 12 shows that for this particular ink, using *n*-propanol as an extraction solvent, the inverse of the ratio of Dye 3/total area of all dyes produces the best aging curve.

Optimum TLC Development

Another important point involves the TLC development procedure. Accurate densitometer readings are not necessarily dependent on completely resolved dye components. It is more

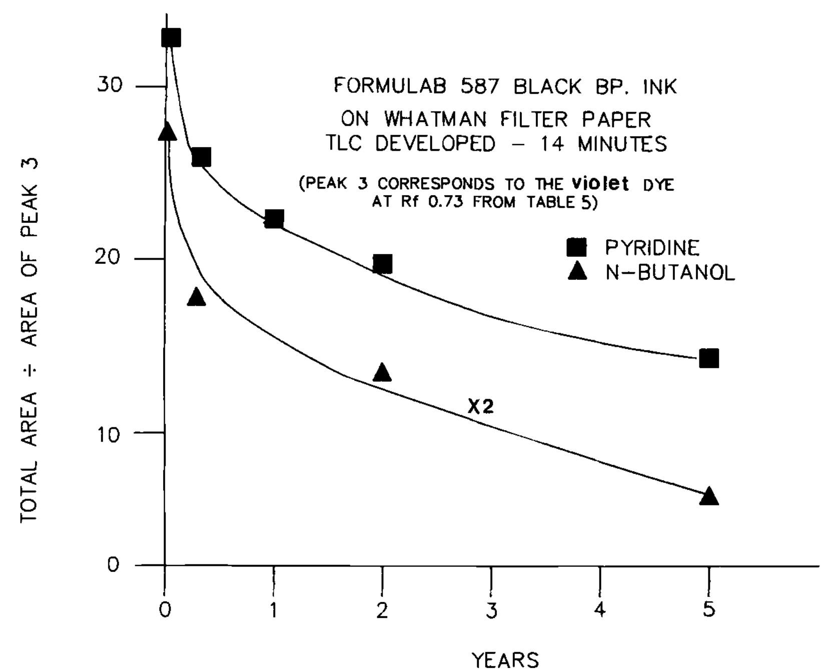

FIG. 11—*Aging curves (strong versus weak solvents).*

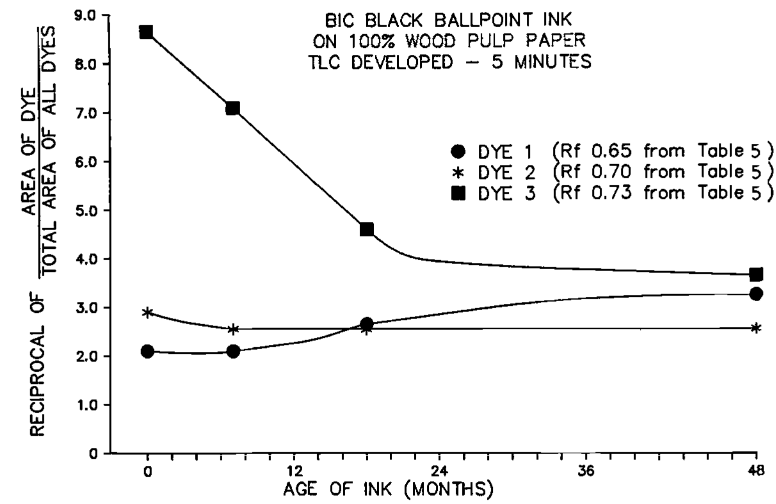

FIG. 12—*Aging curves using various dye ratios.*

important that dye bands be concise and close together on the plate. Therefore, TLC development time should be the minimum time required to separate the dye components and prevent their diffusion on the plate (Fig. 13). Figure 14 shows how the TLC plate looks when densitometer measurements are made by the mass-dependent and mass-independent methods of determining relative age of ink. Table 5 shows the colors of the dyes separated from the inks tested by TLC in this study, along with their corresponding R_f values.

In actual casework situations, it is possible to use both the mass-dependent and the mass-independent ratio methods to estimate the age of questioned ink. The inks spotted on the TLC plates can first be scanned on the densitometer to prepare an age versus amount extraction curve. Then the same spots on the plate can be developed in the solvent system described to separate the dye components for dye ratio calculations. The mass-dependent procedure requires that nearly equal amounts of ink be removed for analysis, however, which requires careful removal of the ink samples from the document.

Limitations

The key limitation of the dye ratio method described is that there is no advance knowledge or assurance that the aging curve will increase or decrease with age and not change in direction (that is, will not be monotonic). This is because the ratios may be of dye components whose aging curves could have different rates. It is necessary to establish all possible aging curves from a set of inks of the same formula and on the same paper for which there are known dates of entry. Some of these limitations are similar to the rate determining methods (*R*-Ratio and *L*th Extraction Time) [*8*]. Induced or accelerated aging procedures to show the direction of the slope can resolve some of these limitations.

Note that it may not be possible to use this mass-independent ratio technique on inks that contain nigrosine. The material produces gray streaks over most of the TLC separation and inhibits accurate densitometer readings of the other dyes present. The ANJA M311 black ballpoint ink is one ink which produced this effect. This problem may be prevented by using a different solvent system for developing the TLC plates. No meaningful aging curve could be obtained with this ink using the solvent system described. The mass-dependent method (measurement of concentration of total unseparated dyes) can still be used in these instances.

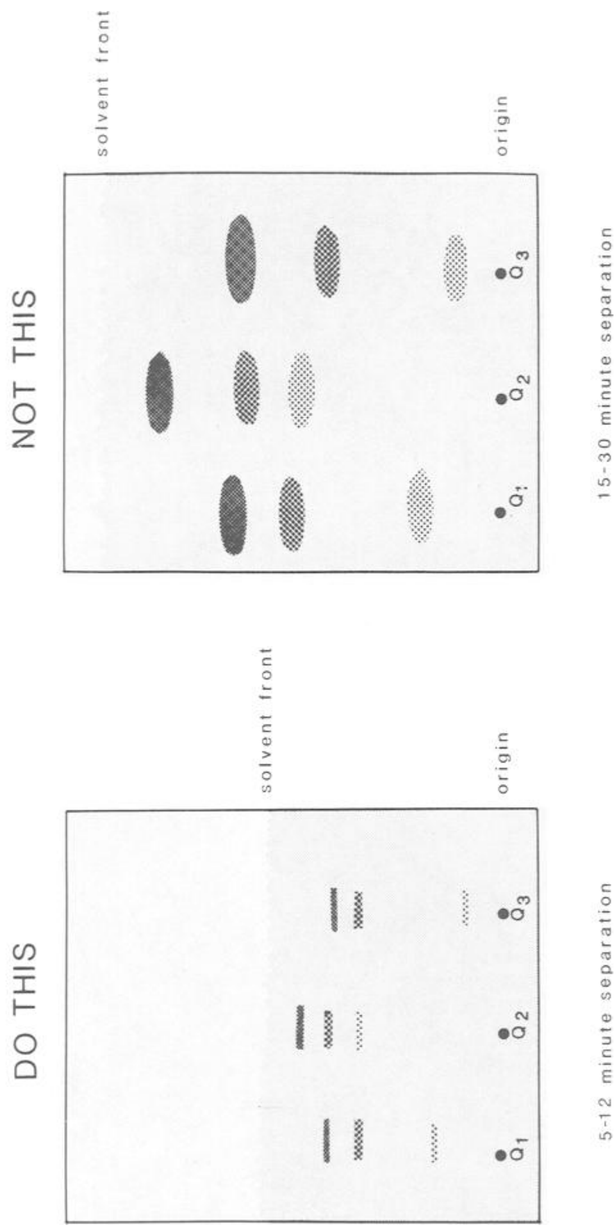

FIG. 13—*Optimum TLC dye separation.*

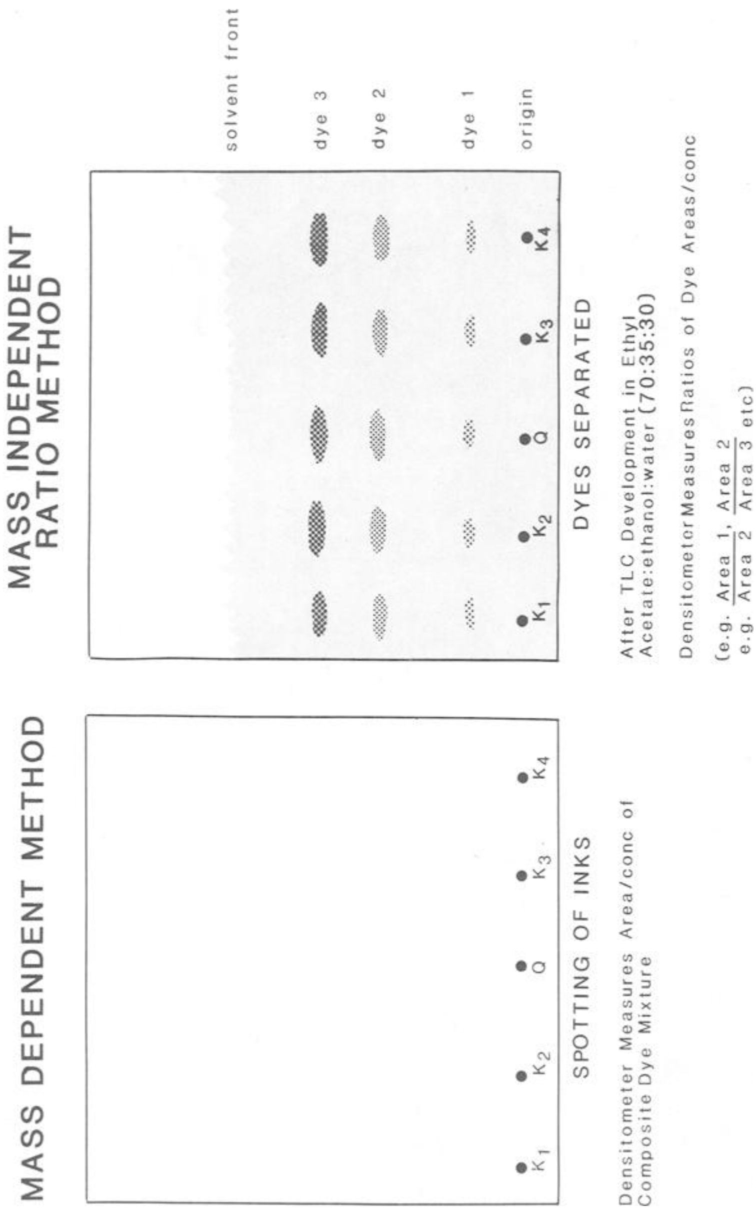

FIG. 14—*TLC procedure.*

TABLE 5—*Dye component and R_f values.*[a]

			BALLPOINT INKS			
Formulab 587 Black	turquoise(0)	violet(0.65)	violet(0.70)	violet(0.73)	violet(0.76)	...
Pilot black	...	violet(0.65)	violet(0.70)	violet(0.73)	violet(0.76)	yellow(0.94)
Bic blue	turquoise(0)	gray-blue(0.48)	violet(0.65)	violet(0.70)	...	...
Bic black	...	violet(0.65)	violet(0.70)	yellow(0.73)	...	...
			NON-BALLPOINT INKS			
Bic (Banana) black	turquoise(0.39)	pink(0.58)	orange(0.70)	...		
Sanford (Sharpie) black	gray ring(0)	light gray(0.68)	dark gray(0.77)	yellow(0.95)		

[a]Whatman silica gel plastic plates developed in ethyl acetate : ethanol : water (70 : 35 : 30) for 15 min.

Conclusions

A simple, easy-to-follow, mass-independent procedure is described that under certain conditions can be used to estimate reliably the age of most ballpoint and some non-ballpoint inks. This study demonstrates that for some inks it is possible to estimate the age of ink up to five years after it is written. The inks examined in this study will be analyzed periodically until their aging ceases. These results will be reported later. It may be possible to show that some of these inks will continue aging for over five years.

It was shown that different paper causes different-shaped ink aging curves, which means that only inks on the same paper can be compared for age. As is generally the case, the inks and documents compared must have been stored under the same conditions (heat, light, humidity, and so forth).

The described procedure appears to be less susceptible to error than other previously reported techniques that are based on solvent extraction. Attempts to determine the age of questioned inks are more apt to be successful because there are many different dye ratio combinations to pick from to prepare ink aging curves. The procedure is not free of limitations, however, as described above.

The feasibility of using the described relative aging technique for non-ballpoint inks was established.

Acknowledgments

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Address requests for reprints or additional information to
Richard L. Brunelle
Brunelle Forensic Laboratories
3820 Acosta Rd.
Fairfax, VA 22031