Characterization of Ballpoint Pen Inks by Thermal Desorption and Gas Chromatography-Mass Spectrometry

ABSTRACT: The characterization of ink on paper is of importance for dating and comparing questioned ink entries in forensic document examination. Inks are commonly characterized by their colorant profile that is identified by well-established analytical methods. Numerous ink formulations show identical colorant profiles, though. In order to differentiate inks that are not distinguishable by colorant analysis, a method for the characterization of colorless ink ingredients, namely binders, solvents and additives is necessary. In this paper, we propose a technique for the analysis of colorless compounds in ballpoint inks using direct thermal desorption of the ink on paper followed by chemical analysis of the desorbed volatile compounds by gas chromatography-mass spectrometry. As compared to liquid extraction and subsequent analysis of the extracts, the technique avoids possible contamination risks. Sensitivity is very high due to the enrichment of volatile components by thermal desorption. Even form old samples, the chromatograms obtained by the method enable the determination of binder polymers, solvents and additives. Pure binders as used by ink manufacturers were analyzed for unambiguous assignment of analytical results to specific polymers. To prove the practical applicability, we analyzed 121 ballpoint pens, not all having the same colorant profile, and grouped the pens into resin and solvent categories.

KEYWORDS: forensic science, questioned documents, ink, composition, resin, gas chromatography-mass spectrometry, thermal desorption, volatiles, document examination

The determination of the age of a pen stroke on paper is a basic subject in questioned document examination. Ink dating may be performed on a relative or absolute scale. Relative in that an ink is compared with reference entries in the same questioned document, and absolute in that no such references exist for comparison (1). There are two main concepts for an absolute dating of ink entries: (i) "indirect" age determination which involves identifying the ink, associating it with a sample from a reference collection, and determining the date of its market introduction, i.e., searching for an anachronism; and (ii) "direct" age determination which involves measuring age dependent parameters of the ink.

This paper focuses on the first "indirect" concept. This concept requires both detailed chemical analysis of the ink composition under investigation as well as an extensive ink library for comparison being updated regularly. Ballpoint pens are currently the most widely used among all types of pens in our forensic casework. Consequently, we focused this study on ballpoint pens. These inks are composed of organic solvents, binder resins, colorants and additives.

Ink libraries maintained by forensic laboratories are commonly classified by colorant profiles. Association of unknown samples is performed by analysis and subsequent comparison of colorants contained in the ink. For the chemical analysis of colorants, i.e., dyes and pigments, well established analytical techniques are available. For the identification and quantification of organic dyes and pigments in the ink composition, the most common techniques are thin layer chromatography (TLC) (2,3) and high performance liquid chromatography (HPLC) (4,5,6). Raman spectroscopy has been suggested for the analysis of dyes and pigments (7,8,9). Other tech-

niques have been proposed to further differentiate colorants in inks (10,11).

Numerous ballpoint ink formulations contain the same mixture of colorants and cannot be distinguished by colorant analysis. Furthermore, compositional changes in ink formulations are occasionally made by the manufacturers by altering solvents, resins or additives while keeping the colorants unchanged. Therefore, it is highly desirable to analyze the solvents and resins used in inks and to classify our ink libraries further into solvent and binder resin classes. So far, inks not distinguishable by colorant analysis may be differentiated in this way.

Solvents and additives in ink samples can be detected by extraction with an appropriate organic solvent and subsequent analysis of the extract using gas chromatography-mass spectrometry (GC-MS) (12,13,14). Nonvolatile colorless components of ballpoint inks were investigated using solvent extraction followed by TLC analysis (15). However, no attempt was made to identify the resins or resin fragments and to use this information for the differentiation of ballpoint inks. One major problem with the analysis of ink on paper is that the absolute amount of analytes available for analysis is less than 100 ng in a typical microplug sample with a diameter of 1 mm and that the matrix (i.e., paper) concentration is higher than 98% by weight. These conditions make the analysis of solvents and additives more difficult with standard analytical methods. Solvent extraction followed by analysis with GC-MS exhibits possible disadvantages with its concomitant effects of dilution and contamination by the extraction step.

In this paper, we introduce a new and simple method for the chemical analysis of ink solvents and resins directly from the paper without sample preparation and without the risk of contamination. Thermal desorption and simultaneous concentration of volatile components lead to low detection limits with respect to the target analytes. Temperature in thermal desorption is chosen high enough to desorb solvents, additives and polymer fragments for the

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characterization of ink binder resins. On the other hand, it is chosen low enough in order to eliminate paper decomposition and minimize signals from the matrix. A collection of 121 ballpoint pen inks was analyzed and grouped into solvent and resin classes.

Methods and Materials

Instrumentation

Thermal desorption of ink samples on paper was performed in a thermal desorption unit TDS2 with autosampler (Gerstel, Mülheim, Germany). This unit was connected to a KAS2 injector (Gerstel, Mülheim, Germany) with glass wool liner equipped with a liquid nitrogen cooling unit. The injector is part of a gas chromatograph-mass spectrometer (Agilent 6840N/5973N). Samples were placed in preconditioned glass thermal desorption tubes (Gerstel, Mülheim, Germany). Tube and glass wool conditioning were performed by purging with argon (100 mL/min) in a tube conditioner (Gerstel, Mülheim, Germany) at 250°C for 30 min.

Thermal Desorption of Ink on Paper

A sample of ballpoint pen ink on paper was cut out (length: 5 to 30 mm; width: 1 to 2 mm) and placed in a thermal desorption tube. The tube was heated in the TDS2 up to the desired temperature (at 12°C/min). Analytes were collected for 5 min by a flow of helium (40 mL/min) and trapped in the cooled injector at -100° C. Subsequently, the injector was heated (280°C, 60°C/min), the compounds were transferred onto the column and analyzed in the gas chromatograph-mass spectrometer (column: HP5MS, $30 \text{ m} \times$ $0.25 \text{ mm} \times 0.25 \text{ }\mu\text{m}$; carrier gas He, constant flow: 1.2 mL/min; oven program: 45°C, 1 min; 45-100°C at 30°C/min; 100-190°C at 12°C/min; 190-270°C at 50°C/min; 270°C, 3 min). Integration of the phenoxyethanol signal was performed on the extracted ion chromatogram (m/z = 138.10). The identification of chemical compounds in the obtained spectra was achieved by comparison of the mass spectra with a standard mass spectral library (NIST/NIH/EPA mass spectral library). Instrument performance was monitored at the beginning of each batch of samples by analysis of 1 μ L of a standard solution of 10 ng/µL phenoxyethanol (Merck, reagent grade, purity > 99%) and 10 ng/ μ L n-hexadecane (Fluka, standard for GC, purity > 99.8%) in acetone (Merck, p. A., purity > 99.8%) on silanized glass wool (Supelco, pesticide grade) in a thermal desorption tube using the aforementioned analytical conditions.

Results and Discussion

Thermal Desorption of Volatile Components

The main aim of our study was to provide an easily applicable method for the analysis of ink components, especially resins and solvents, and to avoid any sample preparation with its concomitant risks of dilution, loss of analytes and contamination. Accordingly, we decided to develop a method for the thermal extraction of volatile components contained in ink samples on paper. In a preliminary study, we used pyrolysis GC-MS to gather information about binder polymers. It turned out that no useable results are obtained when samples of ink on paper are treated conventionally by flash pyrolysis at temperatures between 700°C and 800°C. The paper matrix produced too many signals rendering the identification of signals originating from the ink impossible. Decreasing the temperature in the pyrolysis oven in combination with a careful instrument setup indicated that selective collection of volatile components of interest might be possible. However, conventional pyrolysis equipment has its low temperature limit at about 400°C, which is still too much for the selective analysis of volatile ink compounds.

This limitation was overcome using standard equipment for thermal desorption. Dynamic thermal desorption is an analytical method used in air analysis and polymer characterization using desorption temperatures between 50°C and 400°C (16,17). Selective collection of many analytes can be achieved by selecting the thermal desorption temperature just low enough. We applied this method to samples of ink on paper and tried to desorb the desired fraction slowly while collecting the volatile components in a cooled trap. Pieces of ballpoint ink on paper $(1 \text{ mm} \times 5 \text{ mm})$ were cut out and placed in a thermal desorption tube. Volatile components in the sample were desorbed thermally and transported by a flow of helium carrier gas into the cooled trap. By choosing an appropriate desorption time, nearly quantitative collection of analytes was achieved. We varied the desorption temperature from 150°C up to 250°C to optimize signal intensity for ink components, especially for phenoxyethanol (PE), which is a major solvent in ballpoint pen inks. It turned out that the desorbed amount of PE per unit time stays constant above a temperature of approximately 170°C. On the other hand, above 250°C signals from the matrix paper start to increase drastically in the chromatogram. A desorption temperature of 200°C for 5 min proved to be the optimum for routine analysis of ink on paper. In Fig. 1, an exemplary chromatogram obtained after thermal desorption is depicted. Signals for ink components are

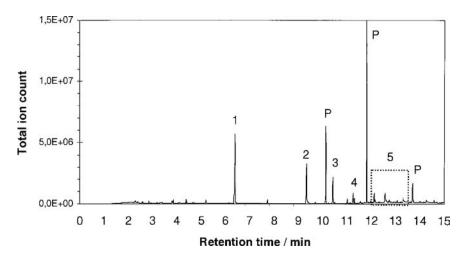


FIG. 1—Chromatogram obtained by thermal desorption and GC-MS analysis of a sample of black ballpoint ink on paper (5 mm length, 1.5 a old). Compounds: 1 = Phenoxyethanol; 2 = Phenoxyethoxyethanol; 3 = Diphenylamine; 4 = Phenazine; 5 = Oligomers of synthetic resin SK; P = Phthalates from sample matrix (paper).

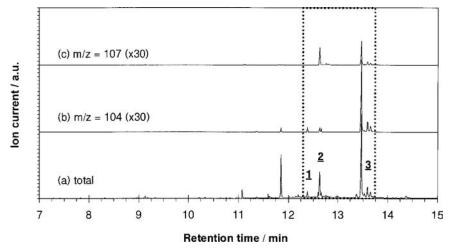


FIG. 2—Characteristic part of a chromatogram obtained by thermal desorption and GC-MS analysis of pure synthetic resin SK. (a): total ion chromatogram; (b), (c): extracted ion chromatograms used for identification of resin SK.

easily identified even in old ink samples. Plain paper samples were analyzed and compared with samples of ink on paper to unambiguously locate and identify ink components in the chromatograms. The signal to noise ratio for ink components was higher than 10:1 with respect to signals originating from the paper matrix. An exception of this rule are ubiquitous plastizisers, such as phthalates, being present in varying amounts in all kinds of paper as well as organic compounds from papers coated with a polymer layer, such as glossy papers for ink jet applications or carbonless copy papers.

A critical aspect in the analysis of ink components by GC-MS, which is hardly addressed in the literature, is the polar character of the main analytes, especially the solvents. For example, phenoxyethanol shows peak tailing in GC-MS analysis caused by strong adsorption to active sites in the analytical gas flow. For this reason, any active sites in contact with the analytes have to be excluded. A regular change of transfer line and deactivated glass wool liner is required, depending on instrument performance. A solution of n-hexadecane in acetone $(10 \text{ ng}/\mu\text{L})$ was chosen as control substance as its recovery in thermal desorption is hardly influenced by active surface sites. To this solution phenoxyethanol $(10 \text{ ng/}\mu\text{L})$ was added and 1 µL was injected onto deactivated glass wool in a thermal desorption tube. This control sample was immediately analyzed in the same way as ink samples on paper. The ratio of the peak integrals of phenoxyethanol versus n-hexadecane is a parameter reflecting instrument performance. The relative standard deviation in this parameter should be below 15% between the tuning intervals of the mass spectrometer. Higher values indicate that a change of instrument components is required. Furthermore, a simple calibration with a solution of the ink solvent phenoxyethanol in acetone (1 to 50 ng/µL, 10 calibration points) was performed to assess the order of magnitude of solvent found in the ink samples. Further standardization procedures were not necessary as the presented analytical method is qualitative in nature.

Characterization of Ink Solvents, Binder Resins, Additives and Dyes

At the forensic institute of the Bavarian State Bureau of Investigation the collection of inks consists of more than 4500 samples and is updated regularly. From these samples, a random choice of 121 ballpoint pens collected in the years 1998 to 2002 was selected and analyzed by the presented method. The total ion chromatograms obtained after analysis of strokes of the selected pens on paper were evaluated using a mass spectral reference library.

It turned out that solvents in ink are easily identified by thermal desorption and GC-MS. As observed previously, the main solvent in most of the analyzed inks was phenoxyethanol, containing phenoxyethoxyethanol (PEE) as minor impurity in some cases (12,18). This impurity can be a major component in old samples of ink on paper as the evaporation rate of the impurity is much lower as compared to phenoxyethanol itself. Many inks contain additional solvents for the adjustment of the writing characteristics of a ballpoint pen. Benzyl alcohol, 2-ethylhexanol, and N-methylpyrrolidone are typical examples for these additional solvents.

Similarly to the analysis of polymers by pyrolysis GC-MS, the identification of binder resins in the ink samples with thermal desorption GC-MS was performed by determination of characteristic, volatile polymer components. For the unambiguous assignment of characteristic components to each binder polymer, thermal desorption analysis of 24 pure binder polymers as used by ink manufacturers was performed. Information about typical monomers and oligomers with characteristic ion fragments in the mass spectra was obtained. Figure 2 shows the chromatographic pattern obtained by thermal desorption and GC-MS analysis of pure synthetic resin SK. The characteristic oligomers identified by their ion fragments m/z =104 and m/z = 107 are marked (number 1 to 3). In Table 1 the main types of pure binder resins and their commercial names are summarized. Table 1 also shows the chemical structures or the names of the main monomers or oligomers and characteristic m/z values observed in the MS-spectra obtained after thermal desorption. The main binder resins used in the selected ballpoint pen inks are based on acetophenone-formaldehyde (AF) resin, cyclohexanoneformaldehyde (CF) resin, and various alkyd resins.

A routine data analysis procedure for the identification of the main binder resin(s) in a ballpoint pen ink formulation was established. This procedure uses the sequential extraction of certain groups of ion fragments from the total ion chromatogram. The presence of a certain group of ion fragments at certain retention times indicates the type of the binder resin contained in the ink formulation at issue (see Table 1). The relative integration of the individual signals of each polymer vary from batch to batch and are not characteristic. Figure 3 shows a typical chromatographic pattern obtained for AF and CF resins from ink samples on paper.

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and GC-MS.			
Binder Resin–Chemical Name	Binder Resin–Trade Names (examples)	Characteristic Monomers or Oligomers	Characteristic Ion Fragments (<u>m/z</u> : r.t./min)
Acetophenone-Formaldehyde- Polymer	Synthetic Resin SK	Ph O Ph	<u>104</u> : 12,4; 12,7; 13,5 <u>107</u> : 12,6
Cyclohexanone-Formaldehyde- Polymer	Synthetic Resin N	CH ₂	<u>98</u> : 11,3; 11,5; <u>110</u> : 11,7; 12,0; 12,1 <u>111</u> : 11,6 <u>122</u> : 5,0
Alkyd-Polymer, modified Polyester	Phthalopal NP, Alkydal BG, Rewopal	 Phthalic anhydride Alkohols (Diols, Glycerol, Trimethylolethane, Pentaerythritol) Fatty acids, rosin 	<u>148</u> : 7,5 <u>239</u> : 12–14
Natural Resins	KPM-Resin	Rosin (e.g., Abietic acid)	<u>284</u> : 13,5
Phenol-Formaldehyde		HO	<u>200, 107, 94</u> : 12,4; 12,62

 TABLE 1—Analyzed pure binder resins (chemical name and trade names) and their characteristic monomers and oligomers found by thermal desorption and GC-MS.

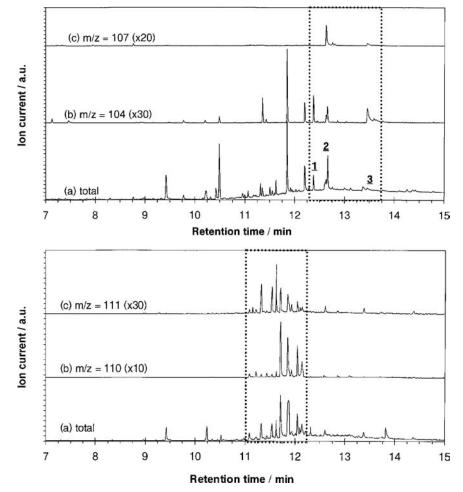


FIG. 3—Part of chromatograms obtained by thermal desorption and GC-MS of ballpoint inks on paper. Top: Ink containing synthetic resin SK as binder. Characteristic signal groups are marked. Bottom: Ink containing synthetic resin N as binder. (a): total ion chromatogram; (b), (c): extracted ion chromatograms used for identification of resin SK and resin N, respectively.

It should be clear that the outcome of the binder analysis by thermal desorption aims at obtaining characteristic information about the ballpoint pen ink under investigation. The purpose of the proposed method is not to identify all the polymers of a binder formulation in an ink. The chosen temperature range for the thermal desorption step limits the obtained data to polymeric components containing volatile monomers and oligomers. Decomposition of the polymers as it is performed by pyrolysis is a minor reaction in thermal desorption. Consequently, the results of the presented analytical method do not include information about polymers having no volatile components.

Together with the information about ink solvents and binder resins, the presence of certain additives was established in many ink samples. These additives are characteristic for an ink sample and can be used for further identification of inks on paper. Typical additives include softening agents, such as phthalates; corrosion inhibitors, such as organophosphates; antioxidants, such as butylated phenols; and surfactants, such as alkylamines. Attention has to be paid to the presence of ubiquitous chemicals present in various amounts in the paper matrix, such as phthalates, being also part of many ink formulations. An unambiguous assignment of such chemicals to an ink sample is difficult. Additionally, in many ink compositions compounds originating from the colorants were found. Diphenylamine, for example, points to the presence of the yellow organic azo dye tropaeoline G (metanil yellow), being a major dye in many black ink compositions. Many black ballpoint pen inks also contain nigrosines, the main structural unit of which is phenazine. Thus, the presence of phenazine in the chromatogram indicates that nigrosine may be present in the ink (see Fig. 1). In this way, the information about colorants in ink obtained by TLC or HPLC is complemented by the results of the analysis by thermal desorption and GC-MS.

The analytical procedure presented is reproducible with respect to qualitative identification of ink compounds. As far as relative amounts of the ingredients are concerned, the method is semiquantitative. A rough indication about the ratio of the ingredients is obtained. It is well known that this ratio may vary with sample age (12,15). A comparison of fresh and old samples may show large differences with respect to amounts of ingredients. In order to study the dependence of the presented method on sample age, we analyzed and compared samples of 10 ballpoint inks of different age. With relative amounts of volatile resin components of old samples (10a) vs. fresh samples (1d) being in the range of 10 to 50%, it was possible to characterize ink resins in old inks exactly in the same way as in fresh inks. However, it turned out that other individual components in ballpoint inks on paper may change or be removed with age. Especially relatively low boiling solvents, such as propyleneglycol, or compounds, which are easily oxidized, such as benzyl alcohol, may not be present any more in old samples. Therefore, attention has to be paid to the nature of the compounds found or expected in the chromatogram, when comparing ink entries of unknown age by analysis of their volatile ingredients.

The type of paper the ink trace is written on, does not influence the obtained results concerning the binder polymers, solvents and other characteristic ink compounds significantly, except that volatile compounds present in the paper matrix also appear in the chromatogram. The presence of such background peaks in the analysis depends on the storage conditions of the questioned document, as paper is capable of absorbing volatile compounds from the environment. Therefore, a blank measurement of plain paper matrix is mandatory for the detailed analysis of ink compounds directly from paper.

Ink Classification

Samples of ink on paper from the above mentioned group of 121 ballpoint pen inks chosen randomly from our ink collection were analyzed and evaluated by the presented method. Subgroups were formed with respect to binder polymers, solvents, additives, and colorant residues. As far as binder polymers are concerned, it turned out that 70 out of 121 pens showed signals for AF resin only, 19 contained CF resin only, 11 had alkyd resins, 4 had phenolformaldehyde resin, 9 had combinations thereof, and 8 out of 121 showed no signals attributable to a pure polymer tested in our study. The 121 pens were further grouped in five solvent classes, depending on the type(s) of solvents found in the ink samples on paper: 25 pens had only PE as solvent; 29 had PE and other solvent(s); 33 had PE and PEE; 32 had PE and PEE and additional solvent(s); and 2 out of 121 pens had no PE but other solvents. For practical documentation we introduced four new fields in the listing of pens of our library in addition to the fields concerning the colorants: one field for identified binder resins (6 subgroups), one field for solvents (5 subgroups), and two fields where additives or rather colorant compounds found in the chromatograms are recorded.

In order to highlight the practical value of the proposed method, a widely used group of black ballpoint pen inks of our collection, internally named 4–19, containing metanil yellow and methyl violet as colorants was analyzed. This group comprises 17 pens with the data of their market introduction ranging from 1991 till 2001. Unfortunately, the pens in this group cannot be differentiated further by analysis of their colorants. The determination of binder resins and solvents is a helpful tool in this case. The analysis of colorant group 4–19 by thermal desorption and GC-MS resulted in 13 subgroups for the 17 pens, thereby increasing the probability for finding an anachronism in the dating of questioned ink entries with the aforementioned colorant composition.

Conclusion

Thermal desorption followed by gas chromatography-mass spectrometry is a valuable tool for the characterization of binder resins, solvents, additives, and colorant compounds from ballpoint pen ink samples on paper. The method requires no sample preparation except cutting out a piece of paper with the writing under investigation. The chosen temperature for the thermal desorption step eliminates signals originating from the paper matrix almost completely while the desired signals originating from ink compounds are obtained in good signal to noise ratios. Comparative measurements with samples of pure binder resins obtained from ink manufacturers led to a procedure for the assignment of signal groups in the total ion chromatograms to individual binder resin classes.

The analysis of samples of a group of randomly chosen ballpoint pen inks on paper from our pen collection showed, that the conventional arrangement of ballpoint pen inks into colorant groups can further be refined by defining groups with respect to binder resins and solvents. These new groups enable more comprehensive discrimination of ballpoint pen inks being indistinguishable by common colorant analysis.

Our future efforts will focus on building up an exhaustive information database about binders and solvents with respect to all the samples of our ink collection. Particular attention will be paid to the identification of binders and solvents in ink types other than ballpoint pen inks. Additionally, the presented method will be applied for investigating the aging behavior of ballpoint inks on the basis of the content of volatile components.

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