

TECHNICAL NOTE

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The Analysis of Fire Debris for the Presence of Propan-2-ol Using Dynamic Headspace Concentration and Gas Chromatography with Flame Ionization Detection

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ABSTRACT: We describe a simple procedure for the identification of propan-2-ol in fire debris. Vapors collected from samples of debris at elevated temperatures, were subsequently dissolved in water prior to being analyzed by headspace gas chromatography with flame ionization detection.

KEYWORDS: forensic science, propan-2-ol, fire debris, gas chromatography, accelerant, arson

The analysis of fire debris for the presence of hydrocarbon accelerant residues is common in forensic science laboratories. A range of methods and techniques based essentially on gas chromatography have been reported in the literature (1–6). The volatility of common accelerants, along with the high proportion of carbon and hydrogen atoms associated with the intrinsic compounds makes gas chromatography with flame ionization detection the method of choice for many investigators.

Sampling of fire debris for common fire accelerants such as gasoline and petroleum distillates including kerosene and diesel fuel is usually performed by passive or dynamic adsorption/desorption. High molecular weight compounds associated with these types of accelerant have relatively low vapor pressures, hence, sampling at an elevated temperature (circa. 100°C) is necessary.

The presence of a fire accelerant in a debris sample is confirmed by visual comparison of the sample chromatogram with standard accelerant chromatograms obtained using identical instrumental conditions. This crude form of pattern recognition forms the basis upon which accelerants such as petroleum distillates are identified.

The following work illustrates a simple procedure whereby propan-2-ol was identified in a sample of fire debris. Although alcohols are not routinely screened for in fire debris by this labora-

tory, the recovery of a container labeled “Iso-propyl alcohol” close to the scene of a suspected arson necessitated this work.

Experimental

Sampling

Vapor samples were removed from the debris samples contained within a nylon bag, using a hypodermic syringe, after pre-heating at 100°C. The syringe contents were discharged into distilled water (5 mL) in a gas tight vial. Subsequent analysis of the aqueous solutions was performed using a GC/FID instrument coupled to a Perkin Elmer HS40 Headspace Analyzer.

Instrumentation

This instrumental procedure is based upon the method we routinely use for the analysis of alcohols in body fluids.

All analyses were performed using a Perkin Elmer 8700 Gas Chromatograph with dual Flame Ionization Detectors coupled to a Perkin Elmer HS40 Headspace Analyzer. The instrumental conditions outlined are routinely used for the quantitation of ethanol in body fluids (7–8). The headspace analyzer conditions were as follows: Headspace mode-constant; the vials were thermostated at 60°C; thermostat time, 13 min; pressurization time, 3 min; transfer line temperature, 110°C; needle temperature, 90°C; injection time, 0.1 min; nitrogen 40 psi. The gas chromatograph conditions were as follows: Oven temperature, 90°C for 4 min; injection temperature, 180°C (splitless); Detector temperatures, both 250°C; hydrogen, 13 psi; air, 20 psi: Data handling:—PE Nelson 1022X GC Plus computing integrator.

Since a single chromatographic peak would be expected for propan-2-ol, its presence was confirmed using a second packed column with a different stationary phase. The headspace analyte vapor was split and chromatography performed on two stainless steel packed columns as follows: (1) 10% Carbowax 400 on CHROM. WAW. (100–120 mesh) 2 m TX 1/8" × 2 mm SS. (2) 0.2% Carbowax 1500 on Carbopack (80–100 mesh) 2 m TX 1/8" × 2 mm SS. Both columns were prepared by Chrompack (Netherlands).

Crime Samples

Fire debris exhibits including a control packing sample were received into the laboratory in sealed nylon bags. Nylon bags are rou-

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tinely used for the packaging of fire debris by this laboratory, however, this type of material is permeable to simple alcohols. Polyolefin bags are considered more appropriate when the presence of alcohols is suspected.

Each exhibit was individually heated in a convection oven at 100°C for 10 min. A vapor sample was removed from each exhibit by puncturing each bag with a hypodermic needle and removing 40 mL of headspace into a plastic syringe. The contents of the syringe were bubbled into deionized water (5 mL) in a sealed vial (20 mL) through a rubber septum. Deionized water blanks were sampled and analyzed between all case and standard runs.

Materials

An aqueous standard solution of low molecular weight compounds (detailed in Table 1) obtained from the Forensic Science Service, Chorley, England, U.K., was used to evaluate the resolution of both chromatography columns for the analysis of propan-2-ol. Propan-2-ol (HPLC grade) was obtained from Rathburn Chemicals Ltd. Walkerburn, Scotland. Deionized water was prepared in-house using a Millipore Alpha-Q water purification system.

Results and Discussion

Baseline resolution was achieved for all of the components of the standard solution detailed in Table 1 on column 2, however partial overlap of the ethanol and propan-2-ol ($R_s = 0.7$) occurs on column 1. The detection limit for propan-2-ol was calculated as $2 \times 10^{-2} \text{ g l}^{-1}$. The repeatability of the retention time of propan-2-ol was assessed on both columns by performing sixteen replicate analyses of

TABLE 1—Standard solution of low molecular weight compounds.

Compound	Concentration ($\mu\text{L l}^{-1}$)
Ethyl acetate	30
Propan-1-ol	300
Diethyl ether	10
Propan-2-ol	125
Ethanol	225
Acetone	20
Methanol	50
Acetaldehyde	10
Propionaldehyde	5

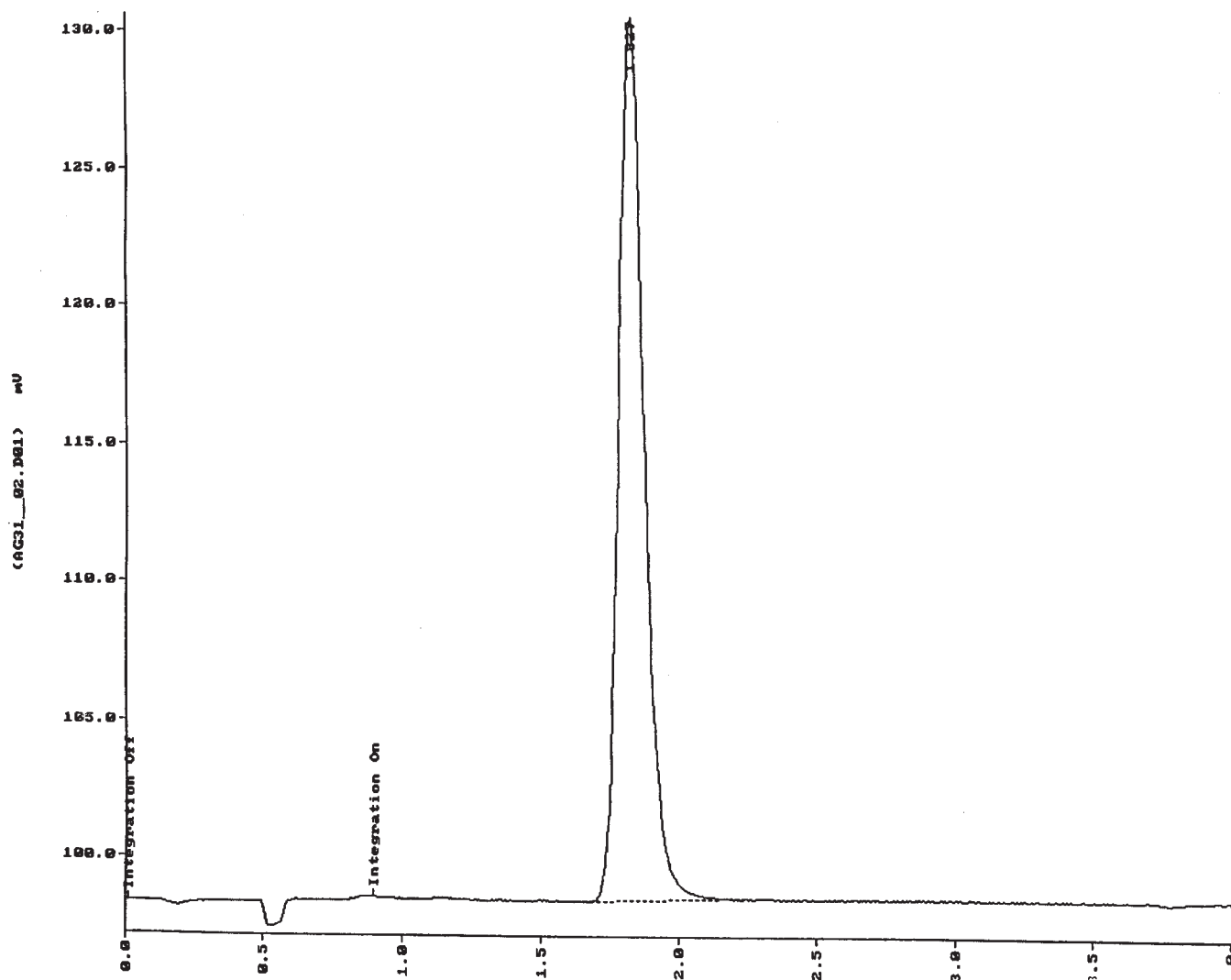


FIG. 1—Gas chromatogram of crime sample—column 1.

an aqueous standard solution of the alcohol of known concentration ($125 \mu\text{L l}^{-1}$).

The mean retention times and associated standard deviations for propan-2-ol on both columns are detailed in Table 2. It was concluded from these results that the chromatographic method outlined, produced reliable data for the identification of propan-2-ol.

Crime Samples

A sample of vapor removed from the crime sample was analyzed on both chromatographic columns (Figs. 1 and 2). The gen-

TABLE 2—Mean retention time, standard deviation and coefficient of variation for propan-2-ol on both chromatographic columns.

	Mean Retention Time	Standard Deviation	Coefficient of Variation (%)
Column (1)	1.828	1.663×10^{-3}	0.091
Column (2)	1.660	8.090×10^{-3}	0.04

erated chromatograms both clearly display a peak with a retention time consistent with propan-2-ol (1.827 min and 1.660 min for column 1 and column 2, respectively).

It is clear from these results, that when present in a sufficiently high concentration, propan-2-ol can be readily detected in fire debris using this method.

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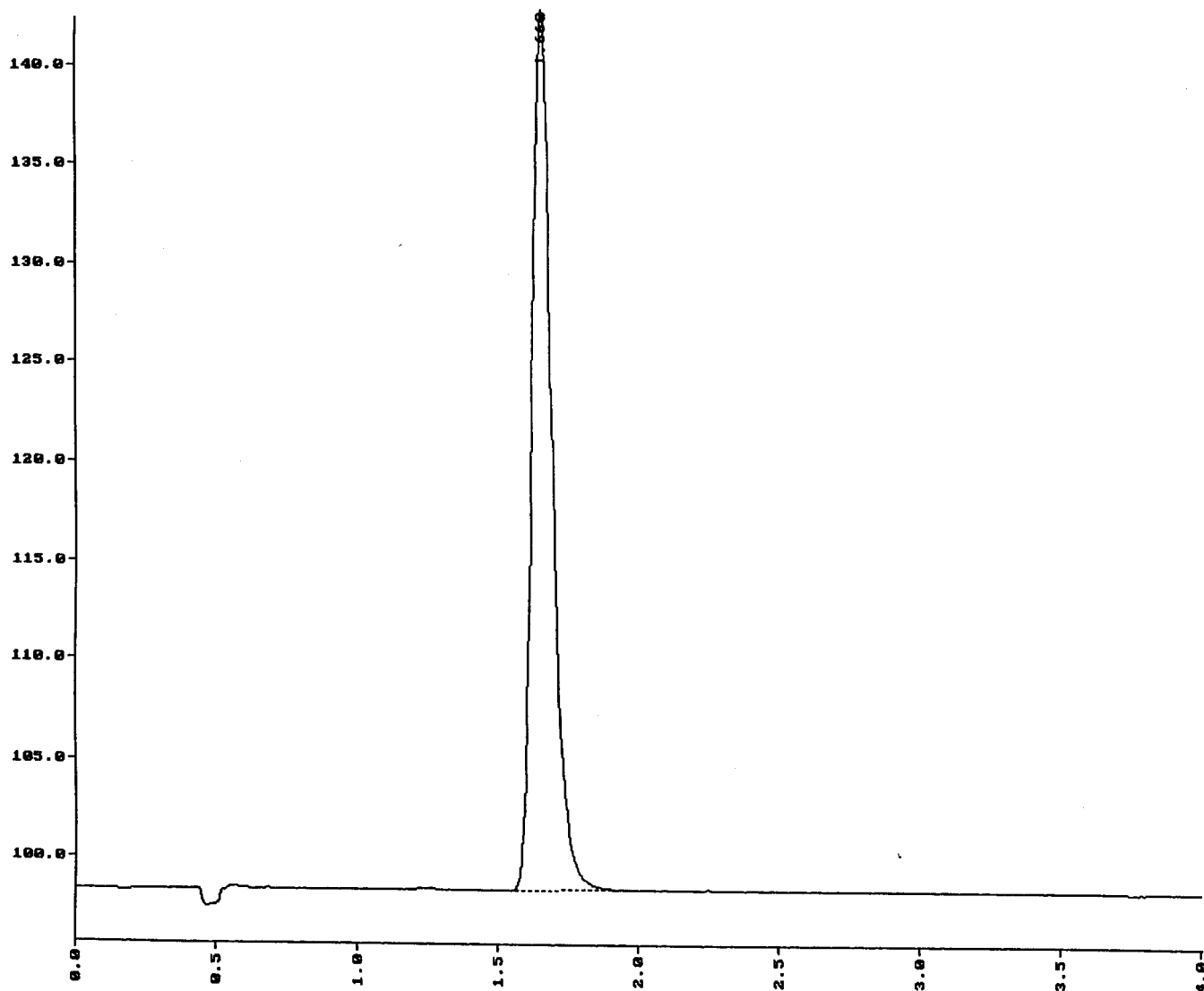


FIG. 2—Gas chromatogram of crime sample—column 2.

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