Journal of Chromatography, 132 (1977) 231–236

CHROM. 9597

WHISKER-WALLED OPEN-TUBULAR GLASS COLUMNS IN GAS CHROMATOGRAPHY

III. APPLICATIONS

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(First received December 5th, 1975; revised manuscript received July 27th, 1976)

SUMMARY

Examples are given of the use of whisker-walled open-tubular columns for separating various mixtures, *viz.*, hydrocarbons, steroids, essential oils, pesticides and fatty acids.

INTRODUCTION

Various aspects of whisker-walled open-tubular (WWOT) columns, including methods of construction and chromatographic performance, have been reported previously[1–3]. In this paper, examples are given of the separations that can be achieved using non-polar, slightly polar and polar stationary phases with a variety of types of mixtures that are important in practice.

EXPERIMENTAL

Columns were drawn from borosilicate glass and provided with a layer of silica whiskers as described previously[2]. The columns were 45–55 m long with I.D. 0.02–0.04 cm. The silica whiskers were deactivated using benzyltriphenylphosphonium chloride[4] when coated with a non-polar stationary phase. When a polar or slightly polar stationary phase was used, prior deactivation was not necessary. Stationary phase was coated on to the whiskers using the dynamic coating method[5]. The columns were then conditioned at room temperature for 24 h, during which time dry nitrogen was passed through them. Finally the temperature was increased at the rate of 1–2°/min to 10–30° above the working temperature of the column and maintained at this level for 24 h (*cf.*, Table I).

The effective plate height, *h*, and the effective number of plates per metre ($N_{eff} \cdot m^{-1}$) were determined in the normal way[3] by injecting a solute for which the mass

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TABLE I

SUMMARY OF THE CHARACTERISTICS AND PERFORMANCES OF THE DIFFERENT COLUMNS USED

Parameter	*1*	*2*	*3*	*4*	*5*
Stationary phase	Squalane	Squalane	OV-101	Dexsil 410	Carbowax 20 M
Column length (m)	44.8	55.4	54	55	48.6
Column radius (cm)	0.012	0.016	0.012	0.014	0.012
Concentration of coating mixture $\left(\frac{\text{weight of stationary phase}}{\text{volume of solvent}}, \%\right)$	5	5	2.5	2.5	5
Conditioning temperature (°C)	130	130	250	270	230
Plate height, H (cm)	0.04	0.04	0.045	0.065	0.055
Effective plate Height, h (cm)	0.09	0.13	0.10	0.15	0.10
$N_{eff} \cdot m^{-1}$	1100	770	950	660	1000
Mean carrier gas flow Velocity, $\bar{u}$ (cm·sec^{-1})	24	14	16	25	24
Mass distribution Coefficient, k	1.85	1.2	1.9	1.9	2.9
Column temperature (°C)	81	81	200	200	220

distribution coefficient, k, varies between 1 and 3, into the separating system at the operating temperature for the column. Data pertaining to the columns are set out in Table I.

A Varian Aerograph Model VA 1800 gas chromatograph was modified to accomodate the column[3]. A flame-ionization detector was used. The splitting ratio at the inlet was 75:1.

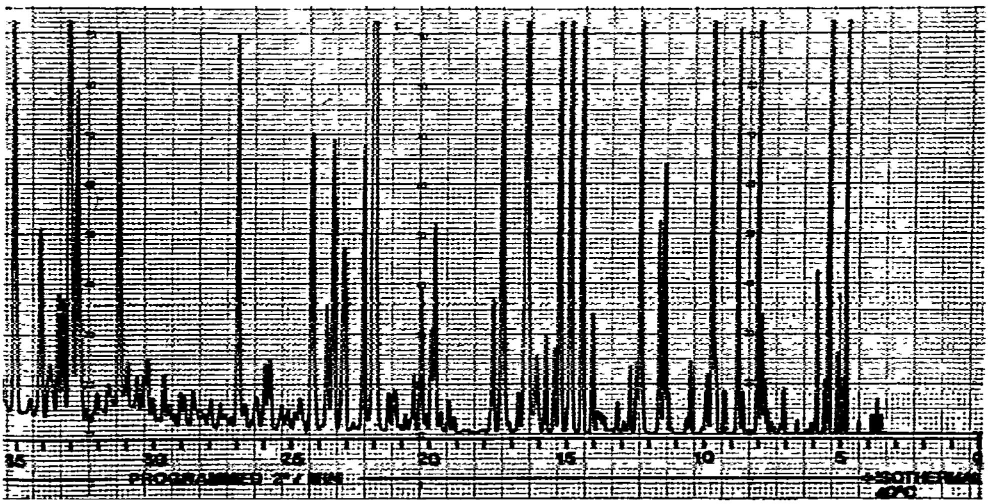

Fig. 1.

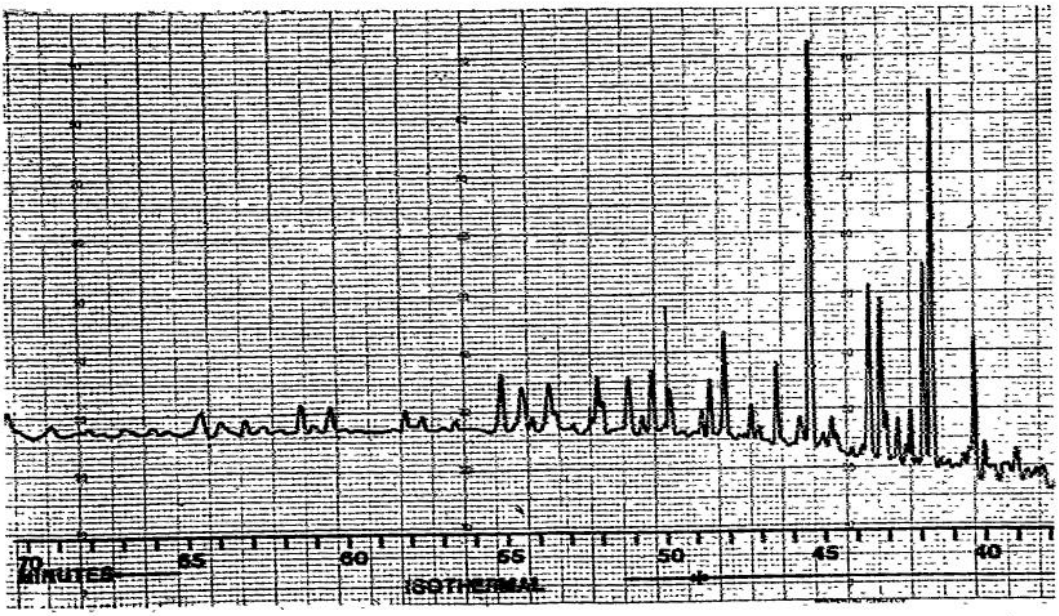

Fig. 1. Analysis of high-octane gasoline prepared synthetically from coal. Column dimensions: 44.8 m × 0.024 cm I.D.; stationary phase: squalane; carrier gas: helium; mean carrier gas flow velocity: 24 cm·sec^{-1}; sample volume injected: 2 μl; column temperature: programmed as shown.

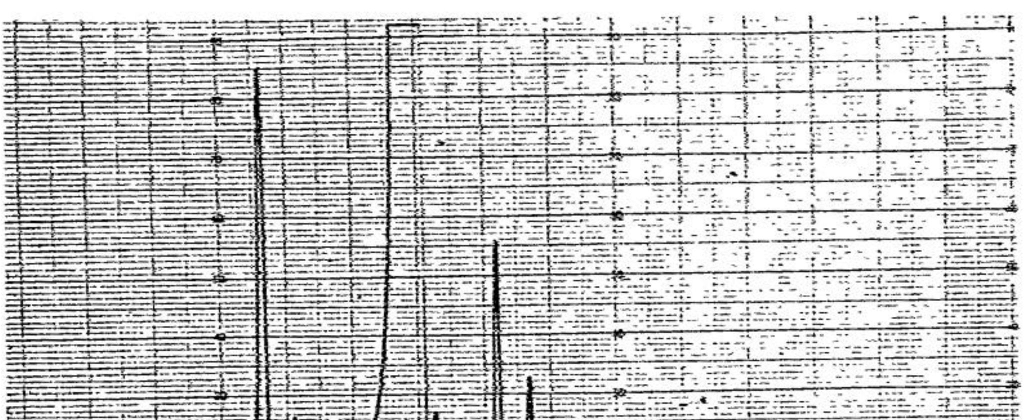

Fig. 2. Analysis of analytically pure *n*-heptane. The peak marked with an arrow corresponds to an amount less than 1 ppm. Column dimensions: 55.4 m × 0.032 cm I.D.; stationary phase: squalane; carrier gas: nitrogen; mean carrier gas flow velocity: 14 cm·sec^{-1}; sample volume injected: 2 μl; column temperature: 85°.

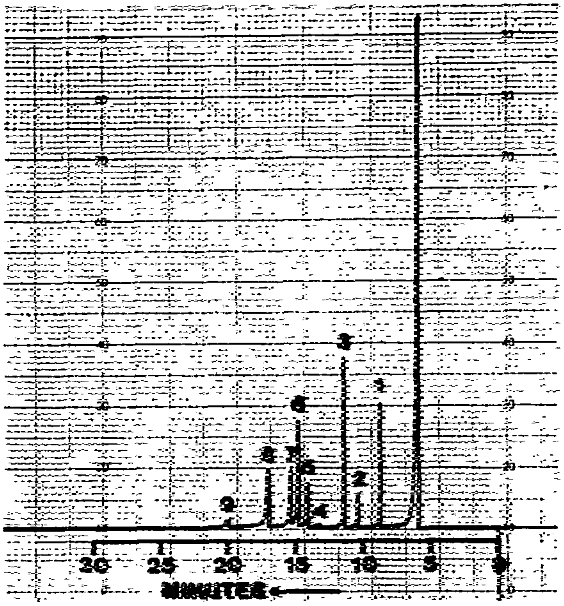

Fig. 3. Analysis of a synthetic mixture of chlorinated pesticides. Column dimensions: 54 m × 0.024 cm I.D.; stationary phase: OV-101; carrier gas: nitrogen; mean carrier gas flow velocity: 15 cm·sec^{-1}; sample volume injected: 1 μl; column temperature: 220°. Peaks: 1 = γ-BHC; 2 = heptachlor; 3 = aldrin; 4 = α-thiodane; 5 = *p,p'*-DDE; 6 = β-Thiodane; 7 = *p,p'*-TDE; 8 = *p,p'*-DDT; 9 = dieldrin.

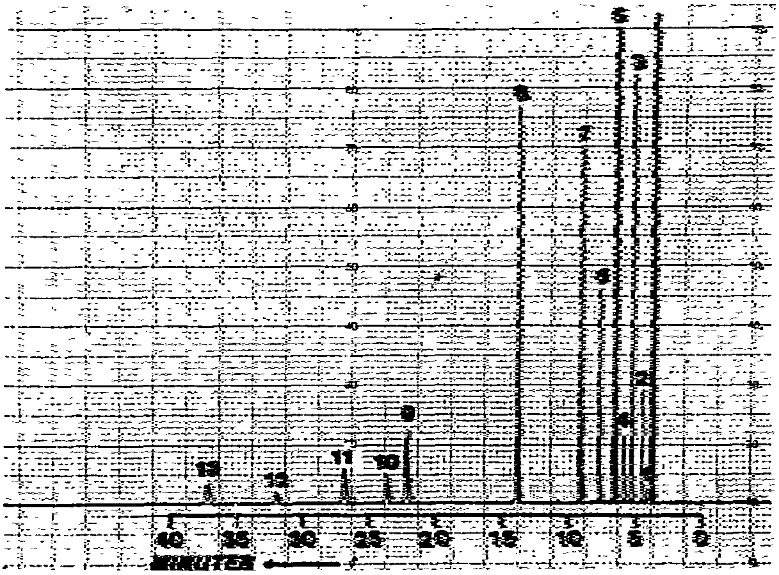

Fig. 4. Analysis of a synthetic mixture of C_6–C_{20} fatty acid methyl esters. Column dimensions: 48.6 m × 0.024 cm I.D.; stationary phase: Carbowax 20M; carrier gas: nitrogen; mean carrier gas flow velocity: 24 cm·sec^{-1}; sample volume injected: 1 μl; column temperature: 220°. Peaks: 1 = methyl caproate (n-C_6); 2 = methyl caprylate (n-C_8); 3 = methyl caprate (n-C_{10}); 4 = methyl undecanoate (n-C_{11}); 5 = methyl laurate (n-C_{12}); 6 = methyl tridecanoate (n-C_{13}); 7 = methyl myristate (n-C_{14}); 8 = methyl palmitate (n-C_{16}); 9 = methyl stearate (n-C_{18}); 10 = methyl oleate (n-C_{18}, 1 double bond); 11 = methyl linoleate (n-C_{18}, 2 double bonds); 12 = methyl linolelaidate (n-C_{18}, 3 double bonds); 13 = methyl arachidate (n-C_{20}).

EXAMPLES OF SEPARATIONS

Chromatograms of various separations are shown in Figs. 1–7.

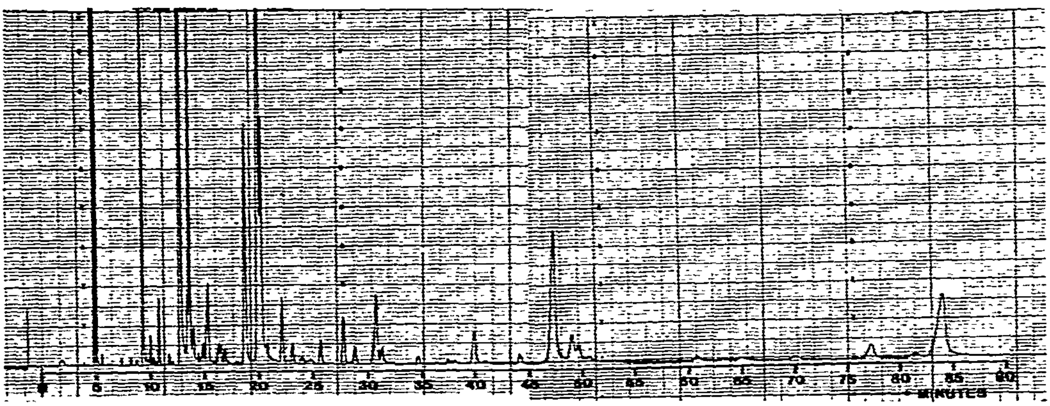

Fig. 5. Analysis of fatty acid methyl esters of a polished mackerel oil. Column dimensions: 48.6 m × 0.024 cm I.D.; stationary phase: Carbowax 20M; carrier gas: nitrogen; mean carrier gas flow velocity: 16 cm·sec^{-1}; sample volume injected: 1 μl; column temperature: 230°.

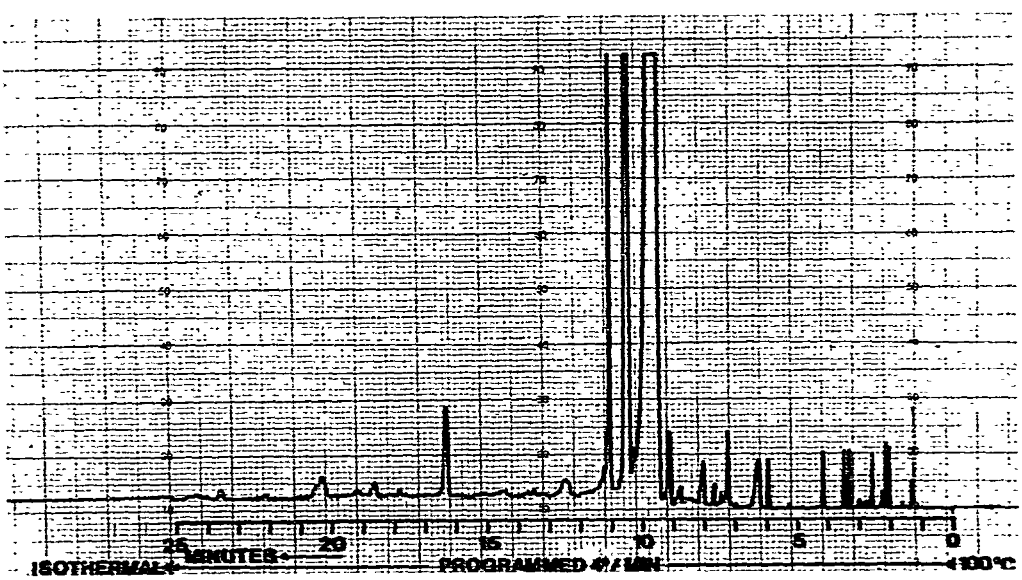

Fig. 6. Analysis of a peppermint oil sample. Column dimensions: 48.6 m × 0.024 cm I.D.; stationary phase: Carbowax 20M; carrier gas: nitrogen; mean carrier gas flow velocity: 24 cm·sec^{-1}; sample volume injected: 1 μl; column temperature: programmed as shown.

DISCUSSION

WWOT columns can be satisfactorily coated with non-polar and polar stationary phases and can be employed to separate many important types of mixtures.

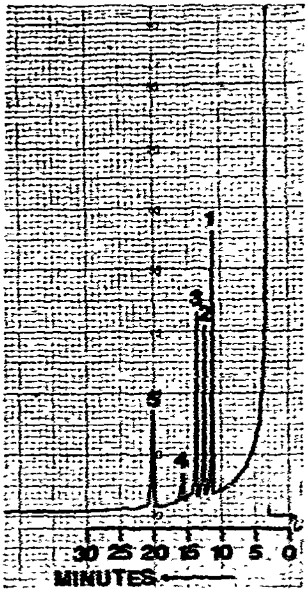

Fig. 7. Analysis of a synthetic mixture of trimethylsilyl (TMS) derivatives of 17-ketosteroids. Column dimensions: 48.6 m × 0.024 cm I.D.; stationary phase: Dexsil 410; carrier gas: nitrogen; mean carrier gas flow velocity: 25 cm·sec^{-1}; sample volume injected: 2 μl; column temperature: 220°. Peaks: TMS derivatives of 1, etiocholanolane; 2, androsterone; 3, dehydroepiandrosterone; 4, 11-ketoandrosterone; 5, 11-β-hydroxyetiocholanolane.

The resolution obtained is at least comparable to that of other types of open-tubular columns. The amount of solute mixture that can be handled by WWOT columns (1–5 μg of a single component) is similar to that with conventional porous-layer open-tubular columns[6].

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