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SIMULTANEOUS GAS CHROMATOGRAPHIC ANALYSIS OF LOWER FATTY ACIDS, PHENOLS AND INDOLES USING A GLASS CAPILLARY COLUMN

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SUMMARY

The simultaneous gas chromatographic separation of a mixture of 14 lower fatty acids, 11 phenols and 7 indoles has been effected by using a glass capillary column. Complete separation of the mixture has been obtained, except for phenol and *o*-cresol and *o*-ethylphenol and 3,5-xylenol whose peaks overlapped, and 2- and 3-methylindoles which were poorly separated. The optimum conditions are as follows: stationary phase, FFAP or PEG-20M; column (20 m × 0.28 mm I.D.) temperature, held for 1 min at 145° (FFAP) or 130° (PEG-20M); column oven, heated at 4°/min from 145 or 130 to 180° (FFAP) or 165° (PEG-20M), maintained at 180 or 165° for 30 min; carrier gas (helium) flow-rate, 0.6 ml/min (FFAP), 1.3 ml/min (PEG-20M); flame ionization detector. The method has been applied to the analysis of lower fatty acids, phenols and indoles in Japanese cigarette smoke, where the peaks were identified by the disappearance method using an alkaline pre-column.

INTRODUCTION

The identification and quantitation of lower fatty acids, phenols and indoles is often required in the organic analysis of foods, cigarettes and cigarette smoke, drugs and automobile exhaust gases, especially in odour pollution analysis. Because these compounds have low odour threshold values, at levels below 1 part per 10^9 in air, the simultaneous analysis of these compounds presents an interesting problem. Direct gas chromatographic (GC) analysis of these compounds at low concentrations has been limited by adsorption and decomposition in the column, ghosting phenomena, the tailing of elution peaks and the separability of many isomeric compounds.

Recently, Robinson[1] reported the GC separation of C_2–C_8 fatty acids on Chromosorb 101, with short analyses times. Okabayashi *et al.*[2] obtained good separations of C_2–C_5 fatty acids by using a FAL-M*, and Di Corcia and co-workers[3–14] separated C_2–C_5 lower fatty acids, phenol isomers, lower aliphatic amines, alcohols and other compounds at the ng level by using gas–liquid–solid chromatography, without adsorption and tailing. Hrivňák and co-workers[15,16] and Barber *et al.*[17] reported the separation of lower fatty acids and phenols by using open-tubular columns

* Polyoxyethylenesorbitan ester + PEG-20M.

containing tricresyl phosphates; capillary columns containing Emulphor O (refs. 18 and 19), squalane[18], Ucon[20] and SF-96 (ref. 21) have also been used for the analysis of lower fatty acids, phenols and indoles, and packed columns of silicone oil DC-550 (ref. 22), SE-30 and 52 (refs. 23 and 24) and XE-60 (ref. 25) and Reoplex 400 (ref. 26) have been used for the analysis of indoles. Unfortunately, when packed columns are employed, the simultaneous GC separation of mixtures of lower fatty acids, phenols and indoles is difficult, because the fatty acids exhibit relatively low boiling points whereas the indoles have higher boiling points and many isomers. In general, the identification of lower fatty acids, phenols and indoles from a complex mixture has been carried out by means of gas chromatography–mass spectrometry (GC–MS). This technique is costly, requires complex instrumentation and is not convenient for on-site environmental pollution analysis.

In this study, glass capillary columns of high resolution were used to achieve the simultaneous GC analysis of low concentrations of lower fatty acids, phenols and indoles, and the identification of these compounds was carried out by the disappearance method using an alkaline pre-column, since these compounds are acidic.

EXPERIMENTAL

Reagents

Fourteen lower fatty acids, 11 phenols, 7 indoles and ethanol were obtained from PolyScience (Niles, Ill., U.S.A.), Aldrich (Milwaukee, Wisc., U.S.A.), Katayama (Osaka, Japan), Tokyo Kasei Kogyo (Tokyo, Japan) and Wako (Osaka, Japan). All of the reagents used were guaranteed or reagent grade chemicals.

Apparatus

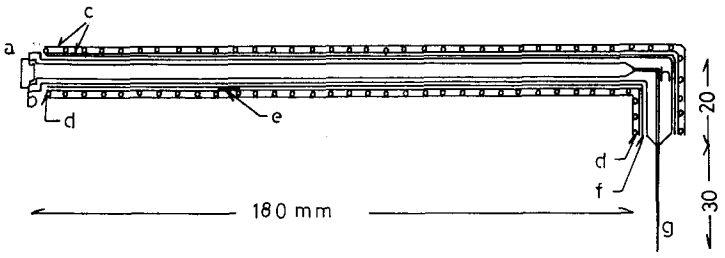
The gas chromatograph used was a Shimadzu Model GC5AP$_5$F (dual columns system) equipped with on-column injection, flame ionization detector (FID) and a digital integrator (Shimadzu Model ITG-2A) for the determination of retention time and quantitative analysis. Two support-coated open-tubular glass capillary (G-SCOT) columns (20 m $\times$ 0.28 mm I.D.) containing FFAP and PEG-20M, respectively, were obtained from Gasukuro Kogyo (Tokyo, Japan).

Chromatographic conditions

The two glass capillary columns were preconditioned at 180° (FFAP) for 3 h, or at 165° (PEG-20M) for 3 h, with a constant flow of helium (*ca.* 1 ml/min) before being connected to the FID. The chromatographic conditions were: (a) stationary phase, FFAP; column temperature, held for 1 min at 145°; column oven, heated at 4°/min from 145 to 180°, maintained at 180° for 30 min and then cooled to 145°; injection port and detector temperatures, 180°; carrier gas (helium) flow-rate; 0.6 ml/min; purge gas (helium) flow-rate, 60 ml/min; splitting ratio, 1:230; air and hydrogen flow-rates for the FID, 1.0 l/min and 50 ml/min, respectively; (b) stationary phase, PEG-20M; column temperature held for 1 min at 130°; column oven, heated at 4°/min from 130 to 165°, maintained at 165° for 30 min and then cooled to 130°; injection port and detector temperatures, 180°; carrier gas (helium) flow-rate, 1.3 ml/min; purge gas (helium) flow-rate, 60 ml/min; splitting ratio, 1:100; air and hydrogen flow-rates for the FID, 1.0 l/min and 50 ml/min, respectively.

Pre-column

Fig. 1 shows a pre-column used for the trapping of samples such as cigarette smoke. It consists of Pyrex glass (18 cm × 4 mm I.D.) packed with Tenax-GC (60–80 mesh)[27–34] and equipped with a nichrome wire for heating and a thermistor for determining the temperature. An alkaline pre-column consisted of 2% potassium hydroxide on glass beads (30–60 mesh): front port, 1.5 cm × 4 mm I.D. plus Tenax-GC (60–80 mesh); needle port, 15.5 cm × 4 mm I.D. The pre-columns were pre-conditioned for 3 h at 220° with a nitrogen flow-rate of 60 ml/min.



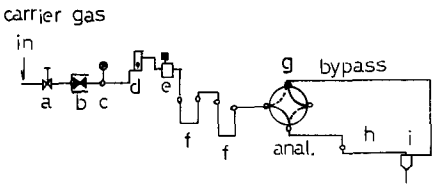


Fig. 1. The trapping pre-column: a = fitted GC injection port septum; b = Pyrex glass tube (4 mm I.D., 6 mm O.D.); c = glass fibre cloth; d = nichrome wire (0.25 mm); e = thermistor; f = aluminium foil; g = syringe needle (0.6 mm I.D.); h = capillary (1.5 mm I.D.). The syringe needle and Pyrex glass tube were fitted by heat treatment.

Fig. 2. Diagram of pre-column injection: a = stop valve; b = pressure controller; c = pressure gauge; d = flow meter; e = mass flow controller; f = molecular sieve 5 A (40 cm × 6 mm I.D. stainless steel); g = four-way cock (one-way stop); h = pre-column; i = GC injection port.

The method of trapping and injecting sample gas was as follows. In the case of standard solutions, the standard solution was injected into the pre-column by use of a microsyringe, and then let into the carrier gas line of the gas chromatograph (Fig. 2). In the trapping of a sample gas (cigarette smoke), the cigarette smoke was trapped in the pre-column at a flow-rate of 0.2 l/min using a vacuum pump. The cigarette used was a Japanese filter-less cigarette (70 mm). After *ca.* 15 min (necessary for the elution of bulky solvent or lower hydrocarbons) the trapped sample was injected into the pre-column, which was set into the carrier gas line of the gas chromatograph, by heating the pre-column with a nichrome wire for 30 sec from room temperature to 200°, maintaining this temperature for 30 min and then cooling to room temperature.

The standard solutions of the reagents were dissolved in water or ethanol to give 10^{-4} mol per 10 ml of solvent. The volume injected was usually 1–6 μl.

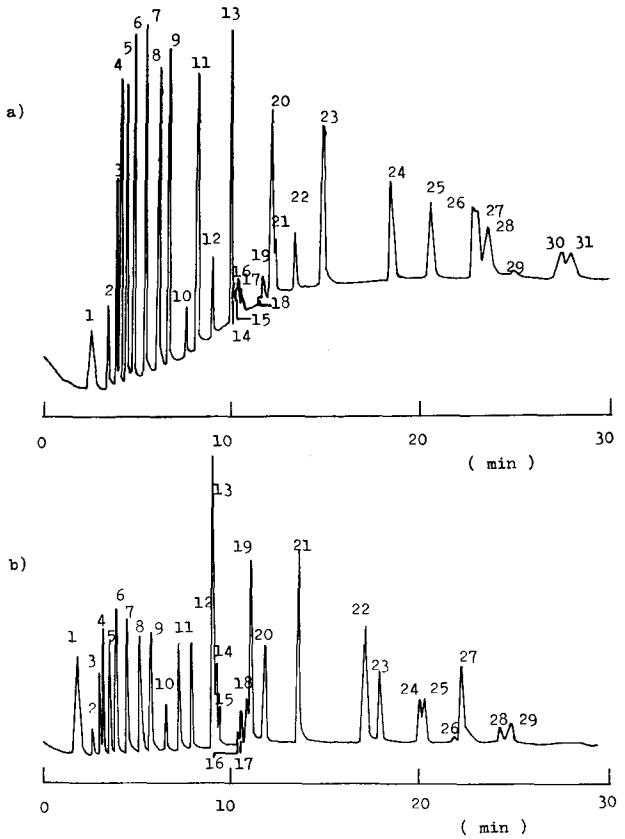


Fig. 3. Typical gas chromatograms of a mixture of 14 lower fatty acids, 11 phenols and 7 indoles obtained by the pre-column injection method using Tenax-GC (18 cm × 4 mm I.D.). (a) G-SCOT glass capillary column (20 m × 0.28 mm I.D.) containing FFAP; other GC conditions as in Experimental, except for FID range 2 (× 0.01 V) and sensitivity 10^3 (× MΩ). Peaks: 1 = solvent (water–ethanol); 2 = CH_3COOH (1.2 μg); 3 = C_2H_5COOH (1.5 μg); 4 = iso-C_3H_7COOH (1.7 μg); 5 = n-C_3H_7COOH (1.7 μg); 6 = iso-C_4H_9COOH (2.0 μg); 7 = n-C_4H_9COOH (2.0 μg); 8 = iso-C_5H_{11}-COOH (2.4 μg); 9 = n-$C_5H_{11}COOH$ (2.4 μg); 10 = 2,6-$C_6H_3(CH_3)_2OH$ (0.5 μg); 11 = n-C_6H_{13}-COOH (2.6 μg); 12 = C_6H_5OH (0.3 μg) + o-$C_6H_4(CH_3)OH$ (0.9 μg); 13 = n-$C_7H_{15}COOH$ (1.4 μg); 14 = p-$C_6H_4(C_2H_5)OH$ (0.3 μg); 15 = 2,5-$C_6H_3(CH_3)_2OH$ (0.2 μg); 16 = p-$C_6H_4(CH_3)OH$ (0.3 μg); 17 = m-$C_6H_4(CH_3)OH$ (0.4 μg); 18 = 2,3-$C_6H_3(CH_3)_2OH$ (0.2 μg); 19 = 1,2-$C_8H_5(CH_3)_2N$ (1.0 μg); 20 = n-$C_8H_{17}COOH$ (1.6 μg); 21 = o-$C_6H_4(C_2H_5)OH$ (0.4 μg) + 3,5-$C_6H_3(CH_3)_2OH$ (0.3 μg); 22 = 3,4-$C_6H_3(CH_3)_2OH$ (0.9 μg); 23 = n-$C_9H_{19}COOH$ (1.7 μg); 24 = n-$C_{10}H_{21}COOH$ (1.9 μg); 25 = C_8H_7N (1.9 μg); 26 = 3-$C_8H_6(CH_3)N$ (1.6 μg); 27 = 2-$C_8H_6(CH_3)N$ (1.5 μg); 28 = n-$C_{11}H_{23}COOH$ (2.0 μg); 29 = 5-$C_8H_6(CH_3)N$ (0.1 μg); 30 = 2,3-$C_8H_5(CH_3)_2N$ (0.9 μg); 31 = 2,5-$C_8H_5(CH_3)_2N$ (1.2 μg). (b) G-SCOT glass capillary column (20 m × 0.28 mm I.D.) containing PEG-20M; other GC conditions as in Experimental, except for FID range 16 (× 0.01 V) and sensitivity 10^3 (× MΩ). Peaks: 1–5, 7–9 and 11 as in (a); 6 = iso-C_4H_9COOH (2.4 μg); 10 = 2,6-$C_6H_3(CH_3)_2OH$ (1.0 μg); 12 = C_6H_5OH (0.6 μg) + o-$C_6H_4(CH_3)OH$ (1.8 μg); 13 = n-$C_7H_{15}COOH$ (2.4 μg); 14 = p-$C_6H_4(C_2H_5)OH$ (0.6 μg) + 2,5-$C_6H_3(CH_3)_2OH$ (0.4 μg); 15 = p-$C_6H_4(CH_3)OH$ (0.6 μg) + m-$C_6H_4(CH_3)OH$ (0.8 μg); 16 = 1,2-$C_8H_5(CH_3)_2N$ (1.0 μg); 17 = 2,3-$C_6H_3(CH_3)_2OH$ (0.4 μg); 18 = 3,5-$C_6H_3(CH_3)_2OH$ (0.6 μg); 19 = n-$C_8H_{17}COOH$ (3.2 μg); 20 = o-$C_6H_4(C_2H_5)OH$ (0.8 μg) + 3,4-$C_6H_3(CH_3)_2OH$ (1.8 μg); 21 = n-$C_9H_{19}COOH$ (3.4 μg); 22 = n-$C_{10}H_{21}COOH$ (3.8 μg); 23 = C_8H_7N (1.9 μg); 24 = 3-$C_8H_6(CH_3)N$ (1.6 μg); 25 = 2-$C_8H_6(CH_3)N$ (1.5 μg); 26 = 5-$C_8H_6(CH_3)N$ (0.1 μg); 27 = n-$C_{11}H_{23}COOH$ (4.0 μg); 28 = 2,3-$C_8H_5(CH_3)_2N$ (0.9 μg); 29 = 2,5-$C_8H_5(CH_3)_2N$ (1.2 μg).

RESULTS AND DISCUSSION

Typical gas chromatograms

Typical gas chromatograms of a mixture of 14 lower fatty acids, 11 phenols and 7 indoles obtained by use of the Tenax–GC pre-column (18 cm × 4 mm I.D., 60–80 mesh) injection method are shown in Figs. 3–5. Fig. 3 shows typical gas chromatograms of a mixture of 32 compounds obtained using FFAP (a) and PEG-20M (b) glass capillary columns. The analysis time of each chromatogram was less than 30 min. The separation of the 14 lower fatty acids was complete in the two liquid phases, but that of the 11 phenols was incomplete, especially for pairs of compounds such as phenol and *o*-cresol and *o*-ethylphenol and 3,5-xylenol. The separation of the 7 indoles was complete except for 2- and 3-methylindole whose peaks were poorly separated. The separation of the two indoles was better on the PEG-20M column than on the FFAP column, but the separation of propionic and isobutyric acids was better on the FFAP column than on the PEG-20M column.

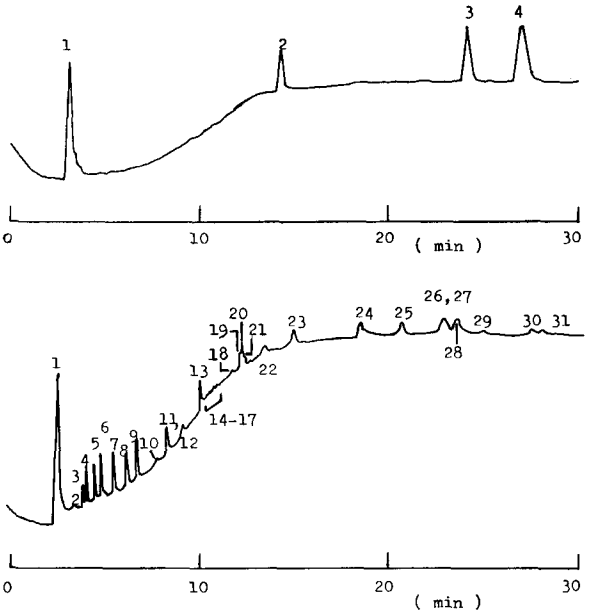


Fig. 4. Typical gas chromatogram of a mixture of 14 lower fatty acids, 11 phenols and 7 indoles obtained by the pre-column injection method using columns of 2% potassium hydroxide on glass beads (1.5 cm × 4 mm I.D.) + Tenax-GC (15.5 cm × 4 mm I.D.). GC conditions and sample size as in Fig. 3a.

Fig. 5. Typical gas chromatogram of a mixture of 14 lower fatty acids, 11 phenols and 7 indoles obtained by the pre-column injection method using a Tenax-GC (18 cm × 4 mm I.D.) column. GC conditions as in Fig. 3a. Peaks as in Fig. 3a but all amounts are one tenth those previously given.

Fig. 4 shows a typical gas chromatogram of a mixture of 14 lower fatty acids, 11 phenols and 7 indoles obtained by use of the pre-column injection method using 2% potassium hydroxide on glass beads (1.5 cm × 4 mm I.D.) plus Tenax-GC (15.5 cm × 4 mm I.D.) and the FFAP glass capillary column. The peaks of 32 compounds

disappeared completely, although three unknown peaks were produced which did not overlap with those of the 32 compounds. It is important that the peaks of the 32 compounds, especially those of the 7 indoles, as well as those of lower fatty acids and phenols, disappeared when the Tenax-GC–2% potassium hydroxide system was used. This system is therefore superior for the identification of the 32 compounds.

Fig. 5 shows a typical gas chromatogram of a mixture of 14 lower fatty acids, 11 phenols and 7 indoles at the 0.1-μg level obtained by use of the Tenax-GC pre-column (18 cm $\times$ 4 mm I.D.). The chromatogram represents at the minimum detectable level of each compound by the present method. As shown in Fig. 5, *n*-butyric acid (a representative odorant of the lower fatty acids) and indole were still detected in the lower limits of 0.17 and 0.19 μg, respectively. Therefore, the minimum detectable quantity of the 32 compounds was *ca.* 0.05 μg, and this method may be applicable to the trace analysis of these compounds.

Fig. 6 shows typical calibration curves for the quantitative analysis of representative compounds, *i.e.*, isobutyric acid, 2,6-xylenol and indoles, obtained using the FFAP glass capillary column with the Tenax-GC (18 cm $\times$ 4 mm I.D.) pre-column. The detector response produced a linear relation in the ranges 0.5–5 μg for 2,6-xylenol, 0.4–20 μg for indoles and 0.5–20 μg for isobutyric acid.

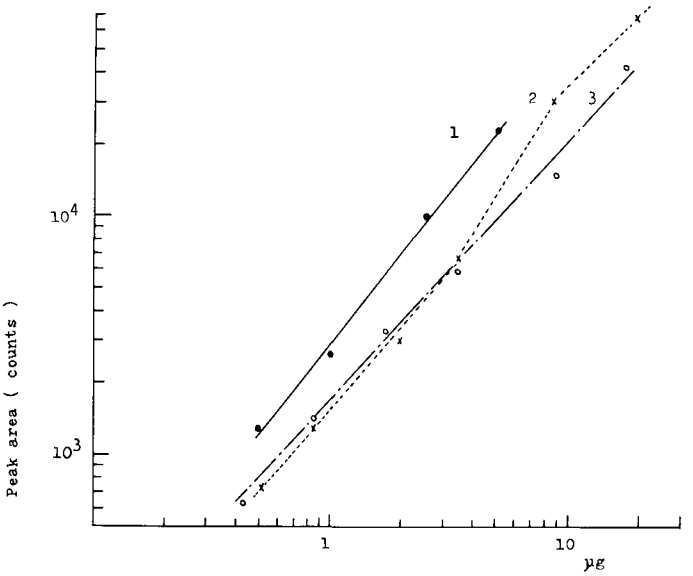


Fig. 6. Typical calibration curves for isobutyric acid (3), 2,6-xylenol (1) and indole (2) obtained using a Tenax-GC pre-column (18 cm $\times$ 4 mm I.D.). GC conditions as in Fig. 3a.

Application to cigarette smoke

Fig. 7 shows a typical gas chromatogram of lower fatty acids, phenols and indoles in the smoke obtained from a Japanese cigarette (10-mm portion of a 70-mm filter-less cigarette). The pretreatment procedures were as described in Experimental. As shown in Fig. 7, at least 75 peaks were recognized and 25 compounds were identified as 10 lower fatty acids, 11 phenols and 4 indoles, *i.e.*, iso- and *n*-butyric, iso- and *n*-valeric, iso- and *n*-caproic, *n*-caprylic, *n*-pelargonic, *n*-capric and *n*-undecanoic

acids, and phenol, *o*-, *m*- and *p*-cresol, *o*- and *p*-ethylphenol, 2,3-, 2,5-, 2,6-, 3,4- and 3,5-xylenol, indole, 2- and 3-methylindole and 1,2-dimethylindole. The quantitative results for the representative compounds expressed in milligrams per cigarette, were as follows: *n*-butyric acid, 0.075; isovaleric acid, 0.09; phenol, 0.13; 2,6-xylenol, 0.01; indole, 0.01; 3-methylindole, 0.05. These results are in reasonable agreement with literature values[35] for the lower fatty acids, but for the phenols were lower than the results of Guerin *et al.*[36], while, the results for the indoles were greater than the values of Hoffmann and Rathkamp[25].

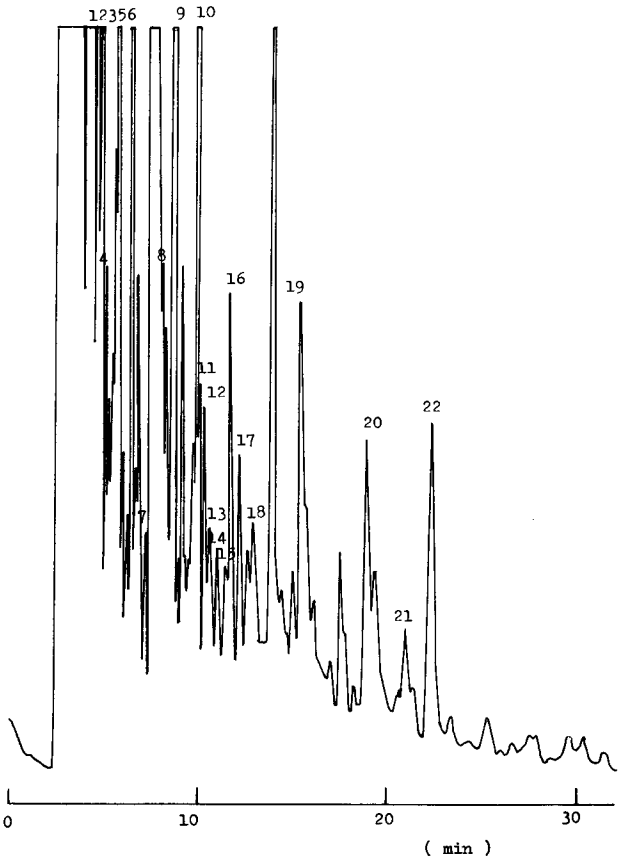


Fig. 7. Typical gas chromatogram of lower fatty acids, phenols and indoles in the smoke obtained from a Japanese cigarette using a Tenax-GC (18 cm × 4 mm I.D.) column. GC conditions as in Fig. 3a. Peaks: 1 = iso-C_3H_7COOH; 2 = *n*-C_3H_7COOH; 3 = iso-C_4H_9COOH; 4 = *n*-C_4H_9COOH; 5 = iso-$C_5H_{11}COOH$; 6 = *n*-$C_5H_{11}COOH$; 7 = 2,6-$C_6H_3(CH_3)_2OH$; 8 = *n*-$C_6H_{13}COOH$; 9 = C_6H_5OH + *o*-$C_6H_4(CH_3)OH$; 10 = *n*-$C_7H_{15}COOH$; 11 = *p*-$C_6H_4(C_2H_5)OH$; 12 = 2,5-$C_6H_3(CH_3)_2$-OH + *p*-$C_6H_4(CH_3)OH$; 13 = *m*-$C_6H_4(CH_3)OH$; 14 = 2,3-$C_6H_3(CH_3)_2OH$; 15 = 1,2-$C_8H_5(CH_3)_2$-N; 16 = *n*-$C_8H_{17}COOH$; 17 = *o*-$C_6H_4(C_2H_5)OH$ + 3,5-$C_6H_3(CH_3)_2OH$; 18 = 3,4-$C_6H_3(CH_3)_2OH$; 19 = *n*-$C_9H_{19}COOH$; 20 = *n*-$C_{10}H_{21}COOH$; 21 = C_8H_7N; 22 = 2-$C_8H_6(CH_3)N$ + 3-$C_8H_6(CH_3)N$.

Fig. 8 shows a typical gas chromatogram of cigarette smoke obtained by use of the column containing 2% potassium hydroxide on glass beads (1.5 cm × 4 mm I.D.) plus Tenax-GC (15.5 cm × 4 mm I.D.). The peaks of the acidic compounds

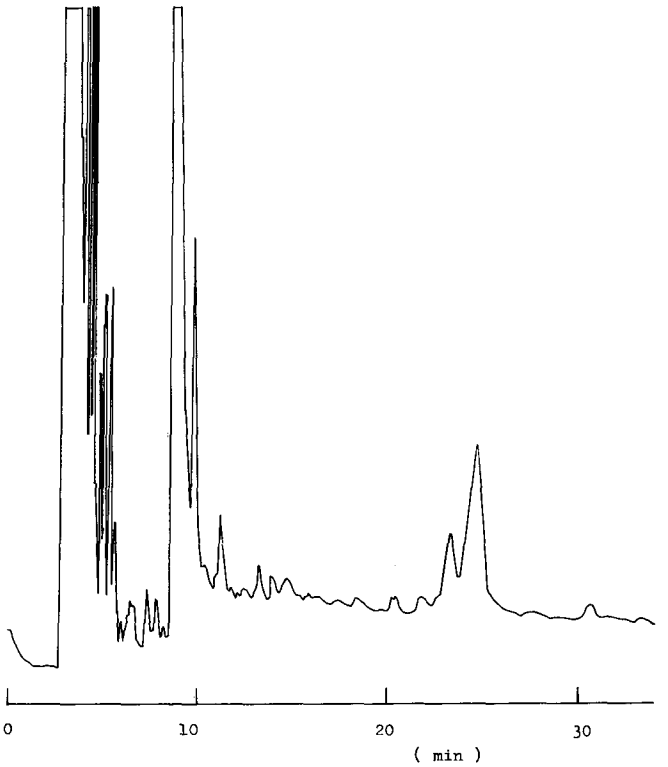


Fig. 8. Typical gas chromatogram of lower fatty acids, phenols and indoles from the smoke of a Japanese cigarette obtained by the pre-column injection method using a column of 2% potassium hydroxide on glass beads (1.5 cm × 4 mm I.D.) + Tenax-GC (15.5 cm × 4 mm I.D.). GC conditions as in Fig. 3a.

in Fig. 7, such as the lower fatty acids, phenols and indoles, respectively, disappeared completely; however, the remaining peaks had longer retention times than on the column containing only Tenax-GC (Fig. 7), *e.g.*, 9 compared with 8 min. The presence of the lower fatty acids, phenols and indoles was confirmed by the disappearance of their peaks on the alkaline column containing Tenax-GC.

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