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EVALUATION OF LIQUID CRYSTAL SMECTIC MESOPHASES FOR GAS–LIQUID CHROMATOGRAPHIC SEPARATIONS

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(Received April 27th, 1981)

SUMMARY

The gas chromatographic properties of the smectic and nematic mesophases of four well-characterized liquid crystals are reported. Selectivity and resolution of anthracene and phenanthrene is greater at column temperatures corresponding to these liquid crystals' smectic A and smectic C phases than at those corresponding to their nematic phases. Peak broadening does not occur in the smectic A or smectic C phases, but is seen in the smectic B phase. Separations of six polycyclic aromatic hydrocarbons and four polychlorinated biphenyls on the liquid crystal columns are compared. The results indicate a continuity of the mechanism of solute separation in the smectic A, smectic C and nematic phases, and demonstrate that these phases are useful in gas chromatographic analyses.

INTRODUCTION

The use of liquid crystalline compounds as stationary phases in gas–liquid chromatography (GLC) is rapidly gaining in popularity[1,2]. They have been successfully used in the GLC analyses of a variety of compounds, including polycyclic aromatic hydrocarbons (PAHs)[3–7], steroid epimers[5,8] polychlorinated biphenyls[10] and methylnaphthalenes[11]. In most cases, liquid crystalline stationary phases have been effectively utilized only in their nematic mesophases. Only a few smectic stationary phases have been studied[4,12–17], and these have produced mixed results. Generally, when compounds having both nematic and smectic mesophases have been studied, it has been found that chromatographic selectivity is significantly worse in the compound's smectic mesophase than in its nematic, despite the lower column oven temperatures that correspond to the smectic structure of these phases. In spite of this, there have been a few successful GLC applications of the smectic mesophase, *e.g.* as GLC phases for the separation and analysis of substituted benzene isomers[12], PAHs[13], insect pheremones[14] and more recently, isomeric olefins[15].

The smectic mesophase exists in several types (identified by letter A, B, C etc.) which correspond to different degrees of order and molecular alignment. The successful application of some smectic liquid crystals in GLC separations suggests that at least some of these smectic types may have potential for use in GC. We have

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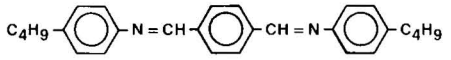
evaluated the GC properties of specific smectic phases of four well-characterized liquid crystals, and compared them with those of corresponding nematic phases.

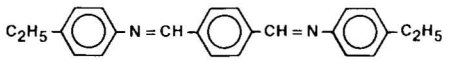
EXPERIMENTAL

Materials

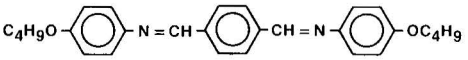
The liquid crystalline compounds were synthesized using published methods[18–21] and purified to give constant transition temperatures (Table I) which matched those previously reported[18 22]. Their structures are given below:



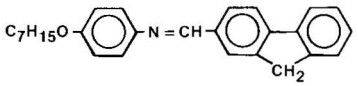
N,N′ terephthalylidene-bis-(4-*n*-butylaniline) (TBBA)[18,21]



N,N′ terephthalylidene-bis-(4-ethylaniline) (TBEA)[18]



N,N′ terephthalylidene-bis(4-*n*-butoxyaniline) (TBBOA)[20]



2(4-*n*-heptyloxybenzylideneamino)fluorene (HBAF)[19,22]

The PAHs were obtained from the International Hydrocarbon Bank (Ottawa, Canada) and the polychlorinated biphenyls from Analabs (North Haven, CT, U.S.A.).

Apparatus and procedure

Mesophase transition temperatures, enthalpies and entropies (Table I) were determined by differential scanning calorimetry with a Perkin-Elmer Model DSC-2 unit.

GC analyses were performed on a Hewlett-Packard Model 7610 gas chromatograph equipped with a flame-ionization detector. Columns packed with the TBBA, TBEA and TBBOA liquid crystals had the dimensions of 6 ft. × 2 mm I.D., while the column containing HBAF was 1 1/2 ft. × 2 mm I.D. Glass columns were used exclusively. The column packing was 2.5 wt. % of the liquid crystal on 100–120 mesh Chromosorb W HP. Chromatograms were on a 1-mV f.s. strip chart recorder using an electrometer setting of 16×10^2. Carrier gas flow (helium, 20 ml/min) was monitored by a calibrated Brooks 5840 Dual GC mass flow controller, while hydrogen and air flow-rates (40 and 500 ml/min, respectively) were measured by a soap bubble flow meter. Sample injection volumes were usually 1–2 μl using a Hamilton 701 N 10-μl syringe.

TABLE I

THERMODYNAMIC CONSTANTS OF THE COMPOUNDS STUDIED

T = transition temperature in °C; ΔH = enthalpy in kcal/mol; ΔS = entropy in cal/mol/°K. C → S_X = crystal to smectic mesophase; S_X → S_Y = smectic phase to next smectic phase; S_Y → N = smectic mesophase to nematic mesophase; N → I = nematic mesophase to isotropic liquid.

Compounds	*Transition*	*T*	*ΔH*	*ΔS*
TBBA	C → S_B	112 (113.0)*	4.4	11.4
	S_B → S_C	144 (144.1)*	0.81	1.9
	S_C → S_A	172 (172.5)*	**	
	S_A → N	198 (199.6)*	0.12	0.25
	N → I	235 (236.5)*	0.18	0.35
TBEA	C → S_B	126 (126)***	3.9	9.8
	S_B → N	149 (149.7)***	2.6	6.2
	N → I	248 (248)***	0.06	0.11
TBBOA	C → S_C	191 (190.6)§	7.0	15.1
	S_C → N	221 (221.2)§	**	
	N → I	295 (295.5)§	0.50	0.88
HBAF	C → S_A	121 (121.5)§§	4.1	10.4
	S_A → N	159 (159)§§	0.19	0.44
	N → I	191 (191)§§	0.29	0.62

* Ref. 21.
** Transition heat is small and diffuse.
*** Ref. 18.
§ Ref. 20.
§§ Ref. 22.

Calculations

Retention times for all solutes are corrected using benzene as the unretained solute. Partial molar enthalpies of solution (ΔH_S) were calculated from the slopes of the linear portions of the graphs of the logarithm of the retention time *vs.* the reciprocal absolute temperature according to the equation of Littlewood *et al.*[23] (slope $= \Delta H_S/R$). The separation factor, α, was determined by the ratio of corrected retention times ($\alpha = t'_{R2}/t'_{R1}$). Resolution was determined by the ratio of the distance between peak maxima and their average peak width at base, as $R = (t'_{R2} - t'_{R1})/0.5\ (W_2 - W_1)$.

RESULTS AND DISCUSSION

The four liquid crystalline compounds used in this study were carefully chosen so that proper evaluation of the GC properties of specific smectic phases could be made. Each of these compounds has at least one well-characterized smectic thermotropic region in addition to a nematic region, so that the GC properties of the smectic and nematic mesophases of each compound could be determined and compared.

Two three-ring PAHs, anthracene and phenanthrene, were used as solutes for the initial evaluation of the columns. Fig. 1 shows the behavior of the corrected retention time as a function of temperature for these two PAHs on the TBBA column. The discontinuity in the curves near the smectic B–smectic C transition temperature (144°C) is typical of the retention behavior of solutes near mesophase transition

temperatures of liquid crystalline GLC phases[1,2,4]. However, no discontinuity in the curves occurs at the smectic C–smectic A or smectic A–nematic transition temperatures (172 and 198°C, respectively); points on the curves through the smectic C, smectic A and nematic temperature ranges fit straight lines with correlation coefficients greater than 0.99. The linearity of these curves in both the nematic and the two least-ordered smectic phases suggests a similarity in the solution properties of these thermotropic regions in GC analyses.

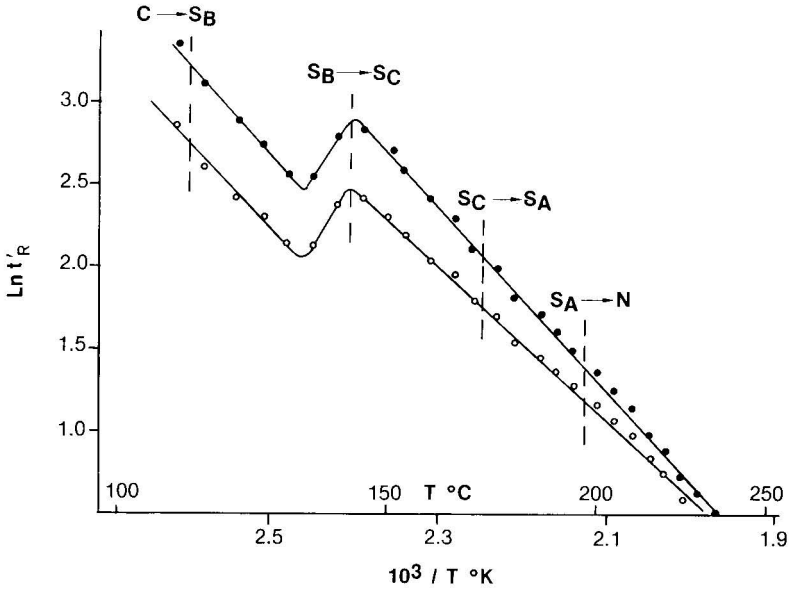


Fig. 1. Retention behavior of anthracene (upper curve) and phenanthrene (lower curve) on the TBBA column in the temperature range 110–240°C. Dashed lines indicate phase transition temperatures.

Partial molar enthalpies of solution (ΔH_1, ΔH_2) of the two PAHs for the various thermotropic regions of each column are listed in Table II. These enthalpies remain constant throughout the smectic C, smectic A, and nematic regions of TBBA, which indicates that the solution properties are constant. In contrast, the ΔH values vary somewhat between the smectic and nematic regions of the TBBOA and HBAF columns. In these two cases however, the difference in the enthalpies of solution for the two PAHs ($\Delta H_1 - \Delta H_2$) does not significantly change between the smectic and nematic mesophases, indicating similar enthalpic selectivity in these modifications. This similarity is reflected in the behavior of the separation factor, α, of the anthracene–phenanthrene system with temperature (Fig. 2). For each of these liquid crystal columns α continues to increase as the column temperature is lowered throughout the nematic, smectic A and smectic C thermotropic regions. Even higher values of α are obtained in the smectic B thermotropic regions of the TBBA and TBEA columns, (Fig. 2a) but these are accompanied by extensive peak broadening, causing substantial drops in resolution at temperatures corresponding to this modification (Fig. 3a). In the other two columns, which do not have a smectic B phase, such peak broadening is not observed until the crystalline state is reached (Figs. 3b, c).

TABLE II

PARTIAL MOLAR ENTHALPIES OF SOLUTION OF ANTHRACENE AND PHENANTHRENE

ΔH_1 = partial molar enthalpy of solution of phenanthracene in kcal/mole; ΔH_2 = partial molar enthalpy of solution of anthracene in kcal/mole.

Column	*Temp. range, °C (mesophase)**	ΔH_1	ΔH_2	$\Delta H_1 - \Delta H_2$
TBBA	145–225 (S_A, S_C, N)	− 9.4	−10.9	1.5
TBEA	145–230 (N)	−13.4	−14.1	0.7
TBBOA	195–220 (S_C)	− 6.5	− 7.5	1.0
	235–265 (N)	−10.2	−11.0	0.8
HBAF	125–145 (S_A)	−12.2	−13.2	1.0
	165–185 (N)	−11.3	−12.3	1.0

* Temperatures where the log retention time *vs.* reciprocal temperature curves are not linear have been omitted.

The transition temperature data in Table I indicates that there is a wide temperature range in which the smectic A and C phases of TBBA overlap the nematic phase of TBEA. Since these two compounds are homologs, and should have similar polarities and molecular alignment characteristics, this allows a direct comparison of the GC properties of smectic and nematic phases in this temperature range. Comparison of the separation factors (Fig. 2a) and resolution (Fig. 3a) of anthracene and phenanthrene in these columns clearly shows the superiority of the smectic A and smectic C phases of TBBA over the nematic mesophase of TBEA. Superiority of the TBBA over the TBEA column in this separation is also indicated by the partial molar enthalpies of solution of the two PAHs for the two columns (Table II). The difference in the ΔH_S values for anthracene and phenanthrene is significantly higher for the TBBA column (1.5 kcal/mole *vs.* 0.7 kcal/mole), indicating higher enthalpic selectivity.

Further comparison of the chromatographic behavior of the TBBA and TBEA columns was made by evaluating the degree of separation of a set of three-ring PAHs at 170°C for each of the two columns. Fig. 4 shows the separation, clearly demonstrating the superiority of the smectic TBBA column. Significantly, while the TBBA column separated all of the components of this mixture, the TBEA column did not resolve 9-methylanthracene and 2-methylanthracene. Relative retention times for a set of polychlorinated biphenyls are shown for the two columns in Table III. Both columns gave similar separation of these compounds, indicating equivalent selectivity of the nematic and smectic modifications in this case.

Relative retention times for the four polychlorinated biphenyls and six PAHs on all of the liquid crystal columns are shown in Tables III and IV, respectively. With column temperatures corresponding to either the smectic C or smectic A phases, and analysis time kept to less than 20 min, adequate separations of these compounds were obtained on all the liquid crystal columns, with the possible exception of the polychlorinated biphenyls on the TBBOA column. Under the conditions used, it appears that the TBBA column is the most versatile. It is significant, however, that good separations were obtained on the short (1 1/2 ft.) HBAF column in its smectic A phase.

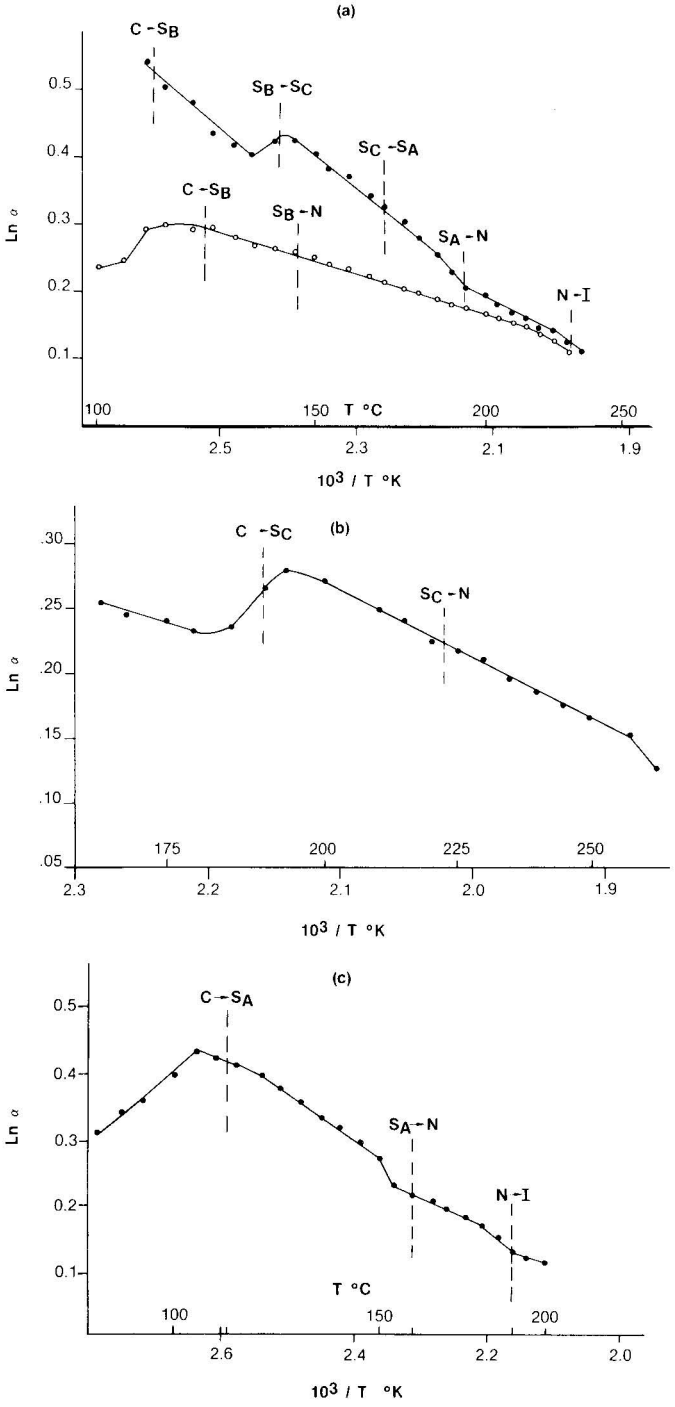


Fig. 2. Dependence of the separation factor (α) of anthracene-phenanthrene on column temperature. (a) TBBA (upper curve) and TBEA (lower curve) columns; (b) TBBOA column; (c) HBAF column.

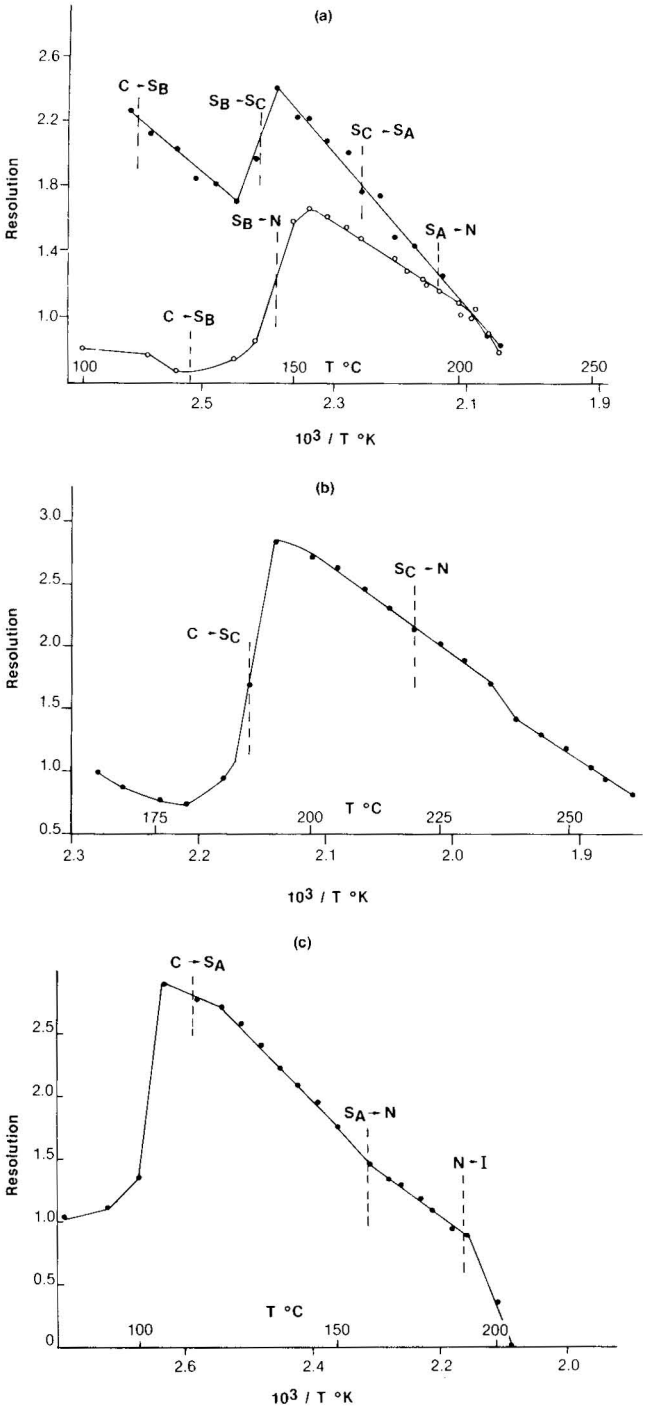


Fig. 3. Dependence of resolution of anthracene and phenanthrene on column temperature. (a) TBBA (upper curve) and TBEA (lower curve) columns; (b) TBBOA column; (c) HBAF column.

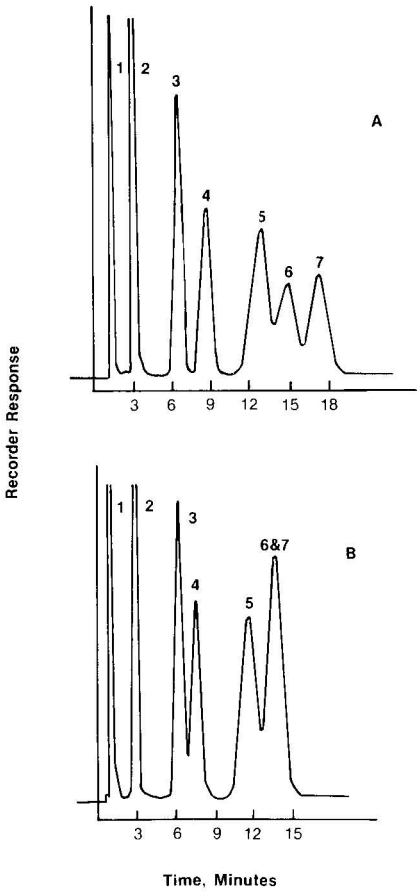


Fig. 4. Separation of three-ring PAHs on the TBBA (A) and TBEA (B) columns at 170°C. Peaks: 1 = solvent; 2 = fluorene; 3 = phenanthrene; 4 = anthracene; 5 = 1-methylphenanthrene; 6 = 9-methylanthracene; 7 = 2-methylanthracene.

TABLE III

RELATIVE RETENTION DATA OF FOUR POLYCHLORINATED BIPHENYLS ON DIFFERENT LIQUID CRYSTAL COLUMNS

TCB = tetrachlorobiphenyl.

Compound	*Relative retention*			
	TBBA at 190°C	*TBEA at 190°C*	*TBBOA at 200°C*	*HBAF at 130°C*
2,5,2′,5′-TCB	1.00	1.00	1.00	1.00
2,4,2′,4′-TCB	1.48	1.55	1.32	1.45
2,3,2′,3′-TCB	1.69	1.70	1.33	1.75
2,3,4,5-TCB	2.13	2.08	1.88	2.41

TABLE IV

RELATIVE RETENTION DATA OF SIX POLYAROMATIC HYDROCARBONS ON DIFFERENT LIQUID CRYSTAL COLUMNS

Compound	*Relative retention*			
	TBBA at 170°C	*TBEA at 170°C*	*TBBOA at 200°C*	*HBAF at 130°C*
Fluorene	1.00	1.00	1.00	1.00
Phenanthrene	2.79	2.92	2.41	1.72
Anthracene	3.83	3.71	3.14	2.49
1-Methylphenanthrene	5.87	6.05	4.50	4.28
9-Methylanthracene	6.85	7.43	5.10	5.22
2-Methylanthracene	8.05	7.43	5.74	5.88

As a result of the problems associated with the use of uncharacterized smectic mesophases in GC analyses in the past, it has been postulated that smectic mesophases offer greater resistance to solute penetration, resulting in inherently lower selectivity in GLC applications[4,17,24]. That this postulate is too sweeping is now indicated by our demonstration of the value of the smectic A and smectic C phases in such analyses, and their superiority over corresponding nematic modifications in certain cases. Moreover, the chromatographic superiority of the TBBA column over that of its ethyl homolog is inconsistent with the widely-held theory that in a homologous series, liquid crystals having the greater nematic temperature range and higher nematic–isotropic transition temperature will have higher chromatographic selectivity[4,25,26].

The enthalpies and entropies of transition (Table I) for the smectic A–nematic, smectic C–nematic and smectic C–smectic A transitions of the liquid crystals used in this study are considerably lower than corresponding values for the smectic B–smectic C, smectic B–nematic and crystal–smectic transitions. In the past, this type of thermodynamic data has been related to liquid crystal mesophase structure[27]. It now appears that such data can be related to GC properties as well. The lower thermodynamic values associated with transitions to smectic A, smectic C and nematic phases indicate a similarity in the structures of the liquid crystalline matrix for these modifications. The chromatographic data presented here indicate an analogous similarity in solution properties, allowing the smectic A and C modifications to be used as GLC phases with equal or greater selectivity than corresponding nematic mesophases.

A major problem still associated with liquid crystal GLC columns is their limited working temperature range, which results directly from the limited nematic temperature range of most liquid crystals. The GC value of the smectic A and C phases of liquid crystals demonstrated in this study indicates that lower column operating temperatures, and hence wider operating temperature ranges, may be achieved with no loss in selectivity through the careful selection and use of liquid crystals having these modifications.

ACKNOWLEDGEMENT

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CONVERSIONS IN CATALYTIC DEAMINATION CALCULATED BY STOPPED-FLOW GAS CHROMATOGRAPHY

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(Received April 14th, 1981)

SUMMARY

By calculating absolute values for the intercepts of the rate equation that describes the deamination of aminocyclohexane on aluminium oxide, the components of a weighted mean rate constant can be found. From these the conversion of the reactant amine to product(s) can be computed, and this is in good agreement with the conversion determined directly by separate experiments. This is an indication of the internal consistency of the theoretical rate equation, derived on the basis of a particular reaction model with many kinds of active sites. The conversions are much less than 100%, showing that a considerable fraction of the reactant is adsorbed on sites which transform it to irreversibly adsorbed products. The conversion to gaseous cyclohexene increases with increasing flow-rate of the carrier gas. Finally, it was found that the effective duration of the stops is very different from the real duration.

INTRODUCTION

In a previous paper[1], the detailed kinetics for the deamination of various amines on aluminium oxide and on porous glass, yielding unsaturated hydrocarbons, were reported. The stopped-flow gas chromatographic technique[2,3] was employed, and the results were consistent with the following reaction scheme:

$$A + S^{(i)} \underset{\text{fast}}{\overset{k_A^{(i)}}{\rightleftharpoons}} \underbrace{A\text{–}S^{(i)} \overset{k_1^{(i)}}{\rightarrow} B\text{–}S^{(i)} \overset{k_2^{(i)}}{\dashrightarrow} D\text{–}S^{(i)}}_{\text{adsorbed species}} \underset{\text{fast}}{\overset{k_D^{(i)}}{\rightleftharpoons}} D + S^{(i)} \qquad (1)$$

according to which the reacting gaseous amine A is rapidly adsorbed on the ith kind of surface active sites with a partition ratio $k_A^{(i)}$, and then gives the adsorbed intermediate B–$S^{(i)}$ with a rate constant $k_1^{(i)}$. The intermediate decomposes, with a rate constant $k_2^{(i)}$, to the adsorbed unsaturated hydrocarbon D–$S^{(i)}$, which is in equilibrium with the gaseous product D, $k_D^{(i)}$ being its partition ratio.

Based on mechanism 1, a theoretical equation was derived, giving the reaction rate, R, as a function of the contact time, t:

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$$R = am\left\{\sum_{i=1}^{n} \frac{k_2^{(i)}k_1^{(i)}g^{(i)}}{\bar{k}_1 - k_2^{(i)}} \cdot \exp\left[-k_2^{(i)}t\right] - \left[\sum_{i=1}^{n} \frac{k_2^{(i)}k_1^{(i)}g^{(i)}}{\bar{k}_1 - k_2^{(i)}}\right]\exp\left(-\bar{k}_1 t\right)\right\} \tag{2}$$

where α is a proportionality constant and m the mass of reactant injected, and $g^{(i)}$ and $\bar{k}_1$ are given by the equations

$$g^{(i)} = \frac{k_A^{(i)}}{1 + \sum_{i=1}^{n} k_A^{(i)}} \tag{3}$$

and

$$\bar{k}_1 = \sum_{i=1}^{n} k_1^{(i)}g^{(i)} \tag{4}$$

The experimental findings on both surfaces studied[1] require that the first summation in eqn. 2 has only two terms. This can happen in three ways: (a) all $k_2^{(i)}$ with $i \geqslant 3$ tend to infinity or, in physical terms, the rate-determining step on these sites is the formation of the intermediate B–S$^{(i)}$; (b) all $k_1^{(i)}g^{(i)}$ for $i \geqslant 3$ are zero, *i.e.*, the amine adsorbs and reacts only on sites 1 and 2; on all other kinds of sites either it adsorbs reversibly [$g^{(i)} \neq 0$] but it does not react [$k_1^{(i)} = 0$], or it does not adsorb at all [$g^{(i)} = 0$]; (c) for $i \geqslant 3$ $k_1^{(i)}g^{(i)} \neq 0$ but $k_2^{(i)} = 0$, *i.e.*, the amine adsorbs and transforms irreversibly to B–S$^{(i)}$, but this does not yield gaseous products.

In all studies published so far in which the stopped-flow technique was employed, the various rate constants k were accurately determined from the slopes of plots of ln R against t; however, for the pre-exponential factors of eqn. 2:

$$a^{(i)} = \frac{k_2^{(i)}k_1^{(i)}g^{(i)}}{\bar{k}_1 - k_2^{(i)}} \tag{5}$$

only *relative* values were determined. These relative values permit us to decide whether possibility (a) above is substantiated or not, but they do not allow us to distinguish between possibilities (b) and (c), because in both instances the pre-exponential factor of the negative term in eqn. 2 is equal to the sum of the values of the other two pre-exponential factors, and this was found to hold for deaminations on aluminium oxide[1]. In contrast, in case (a) the second summation of eqn. 2 contains terms $-k_1^{(i)}g^{(i)}$ with $i \geqslant 3$, which make the summation smaller (absolutely) than the sum of the $a^{(i)}$s of the other two terms. This happens in deaminations on porous glass[1].

The distinction between possibilities (b) and (c) is important, as it affects not the rate constants of the reactions but the extent of *conversion* of amine into the desired product. This can be seen by integrating with respect to t the expression in braces { } on the right-hand side of eqn. 2, between the limits 0 and ∞, obtaining unity in case (b), *i.e.*, 100% conversion, whereas in case (c) the result is

$$\text{conversion} = \frac{k_1^{(1)}g^{(1)} + k_1^{(2)}g^{(2)}}{\sum_{i=1}^{n} k_1^{(i)}g^{(i)}} < 1 \tag{6}$$

i.e., less than 100%. Experimentally, this can be done either by calculating the conversion directly from the chromatogram, or by finding the *absolute* values of the pre-exponential factors $a^{(i)}$, calculating from them $k_1^{(i)}g^{(i)}$ by eqn. 5, and then computing the conversion by means of eqn. 6. The denominator of eqn. 6 is $\bar{k}_1$, according to eqn. 4. The latter method, *i.e.*, through the absolute $a^{(i)}$ values, will demonstrate the internal consistency of the theory underlying eqn. 2, and also its potential for calculating not only rate constants but also conversions through the rate constants.

It is the object of this paper to demonstrate these further potentialities of the stopped-flow technique, by applying the above considerations to a specific case, namely the deamination of aminocyclohexane on aluminium oxide, where the experimental data bear out possibilities (b) or (c) mentioned above.

EXPERIMENTAL

Active aluminium oxide of Brockmann activity II was obtained from BDH (Poole, Great Britain) and sieved to 100–150 mesh. Aminocyclohexane and cyclohexene were purchased from Fluka (Buchs, Switzerland) and were of Purum grade. Nitrogen (99.999% purity) from Linde (Athens, Greece) was used as the carrier gas, after drying it by passage through a tube of 4A molecular sieve.

The apparatus has been described elsewhere[1]. The catalytic column (glass, 1 m × 4.2 mm I.D., filled with aluminium oxide) was conditioned by heating it *in situ* at 543 or 673°K for 24 h under a flow of carrier gas.

Numerical integrations and other calculations were performed on a Hewlett-Packard 9825A desk-top computer connected with a 9872B plotter.

RESULTS AND DISCUSSION

All runs were conducted at 543.2°K. The main product was cyclohexene with a trace amount of benzene. All rate constants reported here refer to the formation of cyclohexene. If h denotes the height of the stop-peaks in centimetres which is proportional to the reaction rate R, the ln (h/cm) *versus* time curves, after a small initial rise, fall off as a sum of two exponential functions. From this behaviour, two rate constants are extracted, as described in detail elsewhere[1]. This corresponds to the first summation in eqn. 2, and the last negative term is responsible for the initial rise.

The effect of the activation temperature of aluminium oxide on the rate constants, as well as the effect of the number of amine injections, were examined first. The catalyst was heated *in situ* for 24 h under a flow of carrier gas, first at 543°K, then at 673°K, and then again at 673°K. Between two successive activations several kinetic experiments were conducted, always at 543.2°K. A t-test of significance was performed on the mean values of the rate constants $k_2^{(1)}$ and $k_2^{(2)}$, calculated from six, seven or nine kinetic runs, for each activation temperature, and this showed that the differences in the $k_2^{(1)}$ values (calculated from the tail of the curves) are not statistically significant at the 10% probability level, whereas the differences in the $k_2^{(2)}$ values are not significant at the 5% or 2% level. This indicates that the effect of the activation temperature on the rate constants is small. The number of injections of the reactant amine on to the catalyst also has a small effect, as judged by the unbiased estimate of the standard deviation of the rate constants for each activation temperature. This was

found to be 8.1–23.4% of the respective mean value. Thus, self-poisoning effects of the catalyst by the reacting amine or the ammonia produced by the reaction are negligible. This conclusion was further confirmed by injecting gaseous ammonia (1 and 5 cm^3 at atmospheric pressure) on to the catalytic column and after 24 h conducting kinetic runs with aminocyclohexane. No significant change in the rate constants was observed.

After these preliminary experiments, the absolute values of the pre-exponential factors $a^{(i)}$ given by eqn. 5 were determined using the equation

$$a^{(i)}_{abs}/sec^{-1} = [a^{(i)}_{rel}/cm] \cdot \frac{(w_{\frac{1}{2}}/sec)\ 1.064}{(m/mol)\ (t_s/sec)\ (S/cm\ sec\ mol^{-1})} \tag{7}$$

$a^{(i)}_{rel}$ are the relative values of the pre-exponential factors in eqn. 2, as determined from the intercepts of ln (h/cm) against time plots, $w_{\frac{1}{2}}$ is the half-width of the stop-peaks, 1.064 is a factor for finding the area under the stop-peaks from their height and their half-width, m the mass of the reactant injected, t_s the duration of each stop and S the response of the flame-ionization detector.

The effective value of t_s does not necessarily coincide with the real time during which the valves supplying the carrier gas are closed, as measured by a watch, and it can be calculated as follows. After injecting 0.5 mm^3 of amine and performing several stops of the carrier gas until the chromatographic signal decays to a negligible height, the total amount of products is given by the integral

$$A = \int_0^{t'} R\,dt = \int_0^{t'} \frac{1.064\ h w_{\frac{1}{2}}}{t_s} \cdot dt = \frac{1.064\ w_{\frac{1}{2}}}{t_s} \int_0^{t'} h\,dt \tag{8}$$

where R is the rate of the reaction, t the time of carrier gas stop and the other symbols have the meanings explained previously. The value of A is found from the total area under the elution curve, which can be measured by a disc integrator. The integral on the far right of eqn. 8 can be found by numerical integration with respect to time of the stop-peak height h above the continuous elution curve. As $w_{\frac{1}{2}}$ is known from the chromatogram, t_s can be calculated, taking care to change A/counts to A/cm sec, *i.e.* to the same units as $\int_0^{t'} h dt$.

Finally, the response S in eqn. 7 was found by injecting known amounts of the pure product into an empty column and integrating the resulting elution curve. This was done for various flow-rate ratios of hydrogen and carrier gas. As expected, the maximum response was found for equal flow-rates of the two gases.

After the above, $a^{(i)}_{abs}$ was found for two carrier gas flow-rates. The results, together with the various physical quantities used in the calculations and the respective rate constants, are given in Table I. An accurate value for the weighted mean rate constant $\bar{k}_1$ could not be determined from the ascending initial part of the experimental curves. The value given in Table I was found using the same experimental conditions but a different method, the so-called reversed-flow gas chromatography[4]. The other two rate constants, $k_2^{(1)}$ and $k_2^{(2)}$, determined by the latter method have about the same values as those found by the stopped-flow technique.

The next two steps were to calculate $k_1^{(i)} g^{(i)}$ from $a^{(i)}_{abs}$ using eqn. 5, and then the conversion by means of eqn. 6. Their values are also given in Table I.

TABLE I

PHYSICAL QUANTITIES CALCULATED FOR THE DEAMINATION OF AMINOCYCLOHEXANE ($4.13 \cdot 10^{-6}$ mol) TO CYCLOHEXENE, AT 543.2 K, ON ALUMINIUM OXIDE ACTIVATED AT 673 K FOR 24 h

Two corrected volume flow-rates of the carrier gas were used, and the stop durations (real) were 60 sec.

Parameter	*Flow-rate, $\dot{V}$ ($cm^3\ sec^{-1}$)*	
	0.191	*0.975*
$10^4\ k_2^{(1)}$ (sec^{-1}), rate constant	1.69 ± 0.05	1.55 ± 0.01
$10^4\ k_2^{(2)}$ (sec^{-1}), rate constant	5.8 ± 0.1	5.8 ± 0.3
$10^3 \bar{k}_1$ (sec^{-1}), weighed mean rate constant*	2.65	
t_s (sec), effective stop duration	15.4	21.8
$w_{\frac{1}{2}}$ (sec), stop-peak half-width	36	9
S (cm sec mol^{-1}), detector response	$5.36 \cdot 10^{13}$	$3.53 \cdot 10^{13}$
$a_{rel}^{(1)}$ (cm), relative intercept	924.7	9657.7
$a_{rel}^{(2)}$ (cm), relative intercept	6612.2	33323.2
$10^5\ a_{abs}^{(1)}$ (sec^{-1}), absolute intercept	1.039	2.910
$10^5\ a_{abs}^{(2)}$ (sec^{-1}), absolute intercept	7.429	10.04
$10^4\ k_1^{(1)}\ g^{(1)}$ (sec^{-1}), calculated from eqn. 5	1.53	4.68
$10^4\ k_1^{(2)}\ g^{(2)}$ (sec^{-1}), calculated from eqn. 5	2.65	3.58
Conversion (%) calculated from eqn. 6	15.8	31.2
Conversion (%) found experimentally	18.9	32.3

* This was found from a separate experiment using the reversed-flow technique (see text).

Finally, the conversion for both flow-rates was determined in separate experiments directly by performing deaminations without stops, finding the area under the product elution curve with a disc integrator and comparing it with the elution curve area of a known amount of pure cyclohexene injected directly on to the same catalytic column. Experiments with pure cyclohexene injected into an empty column showed that the amount of cyclohexene adsorbed irreversibly on the aluminium oxide surface was only 3–5%.

From the results in Table I the following conclusions can be drawn. The reaction model described by eqn. 1 leads to a theoretical rate equation (eqn. 2) which is internally consistent in that it not only explains the time dependence of the rate for the deamination reactions permitting the calculation of rate constants, but it also, through its pre-exponential factors, correlates these rate constants with the total conversion of the reactant to products. This is accomplished by finding the absolute values of the intercepts of eqn. 2 by means of eqn. 7. The calculated conversions are in good agreement with those found experimentally.

From the conversions found or calculated, details of the mechanism emerge, *viz.*, that deamination takes place on two kinds of active sites of aluminium oxide, but a considerable fraction of the reacting amine adsorbs on other kinds of sites and transforms irreversibly to other adsorbed products [case (c) mentioned in the Introduction].

With the help of $a_{abs}^{(i)}$, using eqn. 5, two components $k_1^{(i)}\ g^{(i)}$ can be extracted from the weighted mean rate constant $\bar{k}_1$, and these increase with increasing flow-rate, leading to increased conversions. As pure rate constants, such as $k_2^{(i)}$, do not

seem to change appreciably with the flow-rate, an explanation of why $k_1^{(i)}\, g^{(i)}$ changes is that $g^{(i)}$ increases with increasing flow-rate. This can be due to changes in the apparent values of the partition ratios, $k_A^{(i)}$, because of changes in the column performance with rate.

A final comment concerning the stopped-flow technique is that the effective duration of stops, t_s, is very different from the real duration.

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TRIMETHYLSILYLATION REACTION OF PROSTAGLANDIN-E METHYL ESTER WITH VARIOUS TRIMETHYLSILYLATING REAGENTS

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SUMMARY

Gas chromatographic investigation of the time course of the trimethylsilylation reaction of prostaglandin-E methyl ester (PGE-Me) with trimethylsilylimidazole–piperidine (PIP), N,O-bis(trimethylsilyl)trifluoroacetamide-PIP and other silylating reagents revealed that these reagents do not always give a single product. The reaction products were characterized by gas chromatography–mass spectrometry as the trimethylsilyl derivatives of PGA-Me, PGB-Me, PGE-Me, 9-enol-PGE-Me and 11-piperidyl-PGA-Me.

INTRODUCTION

As part of histochemical studies on marginal periodontal disease, we have examined the situation and extent of localization of prostaglandins (PGs) in human inflammed gingiva with periodontal disease and also the application of gas chromatography (GC) and gas chromatography–mass spectrometry (GC–MS) in this field[1].

Trimethylsilyl (TMS) ethers are widely used in GC and GC–MS analyses of PGs. Recent studies have demonstrated that the reaction of prostaglandin-E (PGE) and its methyl ester (PGE-Me) with certain silylating reagents gave rise to a single peak in the gas chromatogram[2–4].

Nicosia and Galli[2] described a method for the simultaneous dehydration and silylation of PGE-Me to TMS-PGB-Me with a mixture of trimethylsilylimidazole (TMSI) and piperidine (PIP) (1:1). Roselló and co-workers[3,4] also reported a method for the simultaneous one-step derivatization of PGE to TMS-9-enol-PGE with a mixture of N,O-bis(trimethylsilyl)trifluoroacetamide (BSTFA) and PIP (1:1). (Fig. 1). We found that these two methods do not always give a single peak in the gas chromatogram. We therefore re-examined the reaction of PGE-Me with TMSI–PIP, BSTFA–PIP and various other silylating reagents and identified the reaction products by GC–MS.

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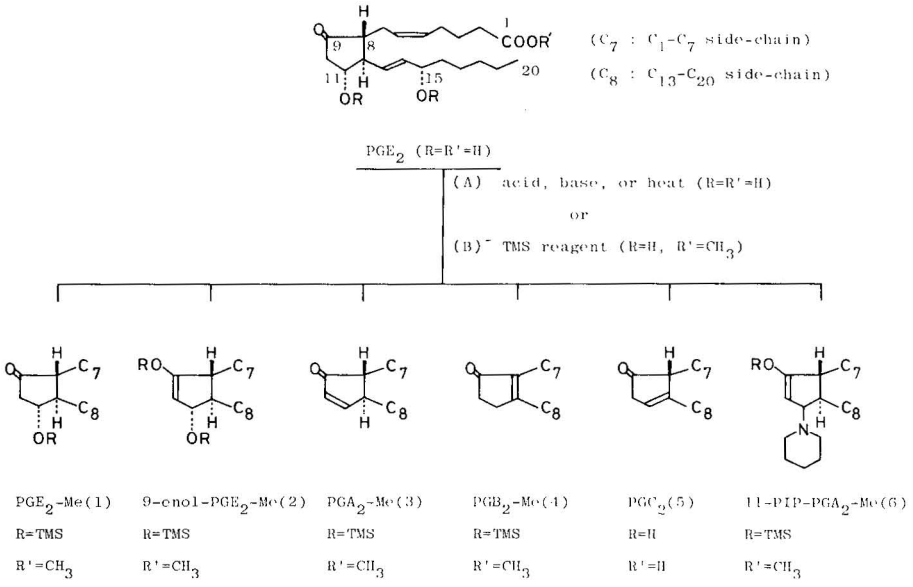

Fig. 1. Derivatization of PGE_2-Me.

EXPERIMENTAL

Reagents

BSTFA (Pierce, Rockford, IL, U.S.A.), TMSI, N,O-bis(trimethylsilyl)acetamide (BSA) (Gasukuro Kogyo, Osaka, Japan), pyridine, PIP, hexamethyldisilazane (HMDS), trimethylchlorosilane (TMCS), acetonitrile (Tokyo Kasei Kogyo, Tokyo, Japan), diethyl ether, methanol, *p*-toluenesulphonyl-N-methyl-N-nitrosoamide (Ishizu Junyaku, Osaka, Japan), PGA_1, PGA_2, PGB_1 and PGB_2 (Sigma, St. Louis, MO, U.S.A.) were commercially available and of standard grade. PGE_1, PGE_2, PGF_1 and PGF_2 were generously donated by Ono Pharmaceutical Co. (Osaka, Japan).

General procedure for the trimethylsilylation of PGA-Me, PGB-Me, PGE-Me and PGF-Me

The prostaglandins (100 μg) were dissolved in methanol (10 μl) and subjected to reaction with ethereal diazomethane (400 μl) prepared from *p*-toluenesulphonyl-N-methyl-N-nitrosoamide (2.14 g) and diethyl ether (30 ml). After evaporation *in vacuo*, the methyl ester was treated with the trimethylsilylating reagent (200 μl). An aliquot of the solution (1–2 μl) was injected into the gas chromatograph with a 5-μl Hamilton microsyringe.

Gas chromatography–mass spectrometry

A Shimadzu GCMS-7000 gas chromatograph–mass spectrometer equipped with a High-Speed MID-PM 9060S multi-ion detector was used under the following conditions: column, glass, 1 m × 3 mm I.D.; packing, 1.0% OV-17 on 60–80-mesh Chromosorb W AW, DMCS (Nishio Kogyo, Osaka, Japan) and 1.5% OV-210 on

80–100-mesh Shimalite W AW, DMCS (Shimadzu Seisakusho, Kyoto, Japan); carrier gas, helium at 30 ml/min; column temperature, 220°C for the OV-17 and 215°C for the OV-210 column; injection port temperature, 260°C; separator temperature, 270°C; ion source temperature, 290°C; ionizing voltage, 20 eV; trap current, 70 μA; accelerating voltage, 3 kV; recorder range, 2 mV; chart speed, 1 cm/min.

Mass spectrometry

Mass spectra were obtained with a Shimadzu GCMS-7000 gas chromatograph–mass spectrometer at 70 eV. The Visigraph was run at 10 cm/min (*ca. m/e* 700).

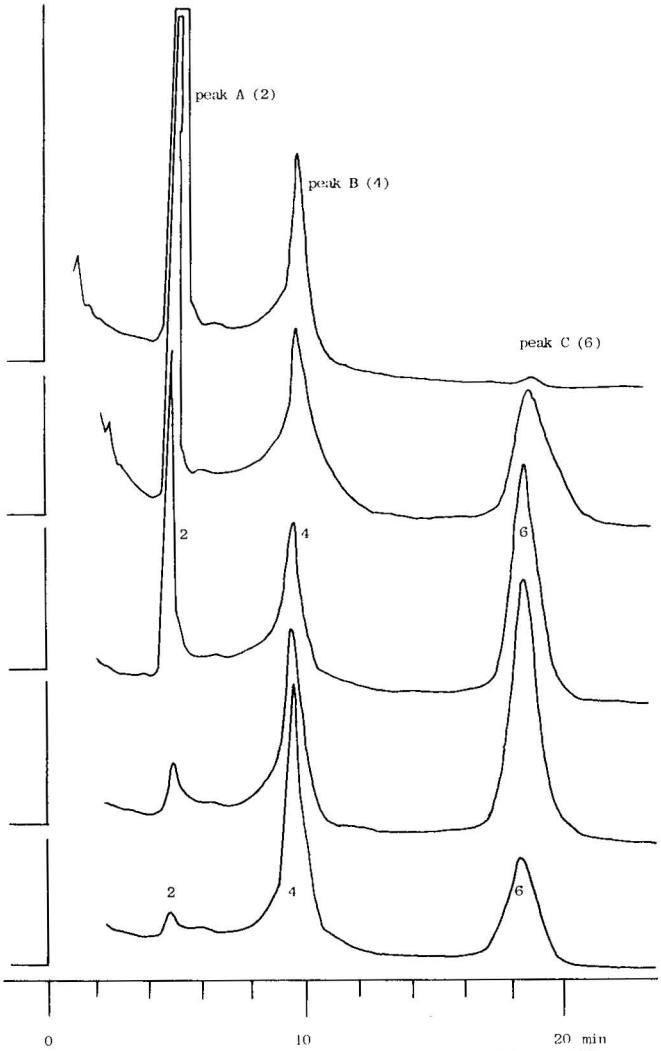

Fig. 2. Time course of the reaction of PGE_2-Me with PIP–TMSI reagent on an OV-17 column (1 min, 20 min, 50 min, 2 h and 5 days, reading downwards).

RESULTS AND DISCUSSION

It is well known that PGE, having an unstable β-hydroxy ketone ring structure, is readily dehydrated to the corresponding PGA, PGB and PGC (R = R′ = H in Fig. 1) by acid, base or heat. The problem arose, especially with seminal fluids, of whether PGA, PGB, 19-hydroxy-PGA and 19-hydroxy-PGB are natural compounds or artefacts[5–7]. Thermal degradation of PGE might be expected under much more severe GC conditions. In general, the ketone function of PGE is protected by the heat-stable alkyloxime formation and a variety of PG oximes have been reported. However, the formation of two geometric isomers of the *syn/anti* type is not desirable from an analytical point of view.

Nicosia *et al.* and Roselló and co-workers reported that the one-step reaction

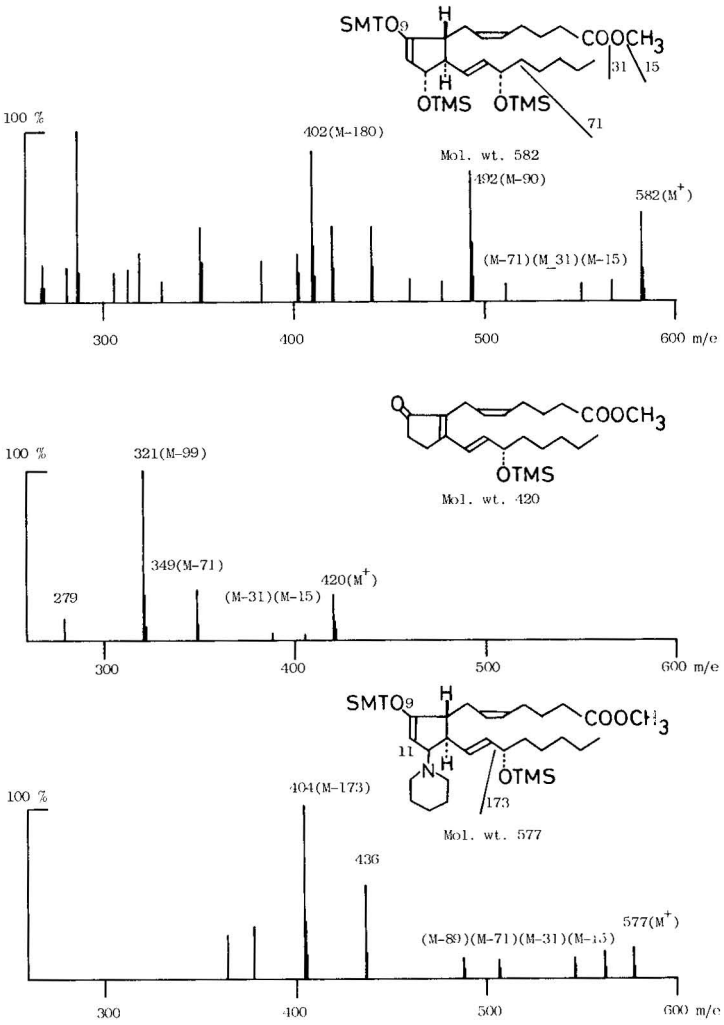

Fig. 3. Mass spectra of peak A (top), peak B (middle) and peak C (bottom) at 70 eV obtained by GC–MS.

of PGE and PGE-Me with TMSI–PIP (1:1) and BSTFA–PIP (1:1) could give a single peak in the gas chromatogram.

We first examined the reaction of PGE_2-Me with TMSI–PIP (1:1) according to the method of Nicosia *et al.* GC–MS analysis of the reaction mixture showed two additional peaks, in addition to TMS-PGB_2-Me (4) described by Nicosia *et al.*

The time course of the reaction is shown in Fig. 2. After 1 min at room temperature, three peaks (A, B and C) with a peak-height ratio of 75:21:1 were observed in the chromatogram. This ratio shifted to 40:15:10 after 20 min, 12:6:8 after 50 min, 2:6:9 after 2 h and 2:20:7 after 5 days. On the other hand, after 5 min at 85°C, the ratio was 2:20:7.

These peaks were finally identified as TMS-9-enol-PGE_2-Me (2), TMS-PGB_2-Me (4) and TMS-11-piperidyl-PGA_2-Me (TMS-11-PIP-PGA_2-Me) (6) by comparison with authentic samples and data from the literature (Fig. 3). This is the first case in which PGE_2-Me has been converted into a PGA_2-Me derivative(TMS-11-PIP-PGA_2-Me) (6) by TMSI–PIP reagent, although PGA has been converted into TMS-11-PIP-PGA by BSTFA–PIP reagent[5].

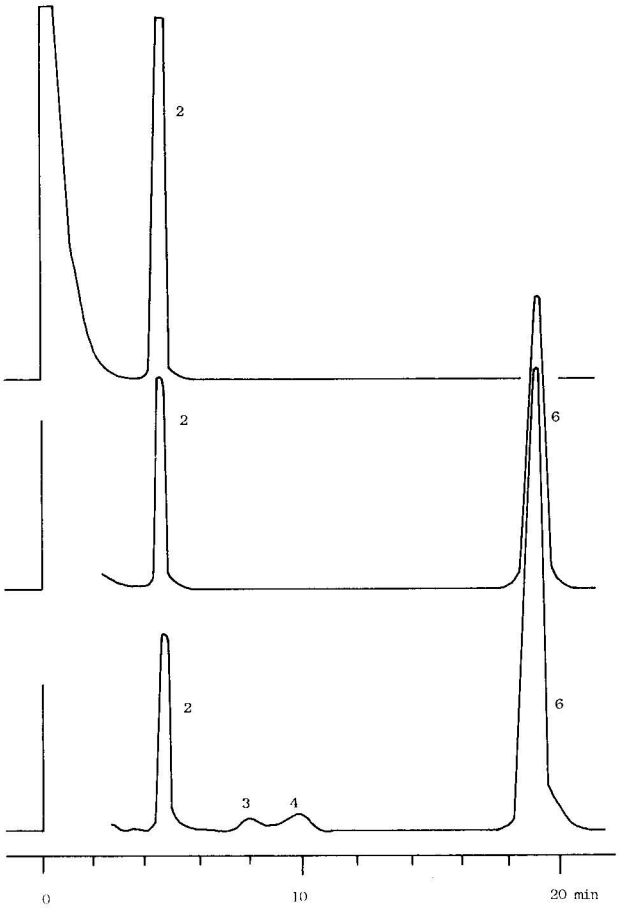

Fig. 4. Time course of the reaction of PGE_2-Me with PIP–BSTFA reagent on an OV-17 column (2 h, 2 weeks and 1 month, reading downwards).

Next, we examined the reaction of PGE_2-Me with BSTFA–PIP (1:1) according to the method of Roselló and co-workers.

The time course of the reaction is shown in Fig. 4. A single peak of TMS-9-enol-PGE_2-Me (2) was observed and reaction product was stable for at least 48 h, as described by Roselló and co-workers. However, an additional peak, which was identified as TMS-11-PIP-PGA_2-Me (6) by GC–MS, appeared after 2–3 weeks. Moreover, two other peaks, identified as TMS-PGA_2-Me (3) and TMS-PGB_2-Me (4), appeared in small amounts after 1 month. The same phenomena were observed with PGE_1-Me derivatives.

Lastly, the reaction of PGE_2-Me with various trimethylsilylating reagents was examined by GC–MS using an OV-210 column.

The results are shown in Table I and Fig. 5. The gas chromatogram of TMS-PGF_1-Me and TMS-PGF_2-Me is also shown in Fig. 5 in order to compare their retention times with those of TMS-PGE_2-Me derivatives. All of the reaction products were identified by comparing their retention times and mass spectra with those of authentic samples and data from the literature.

TABLE I

REACTION PRODUCTS OF PGE_2-Me WITH TMS REAGENTS

Reagent	*Composition*	*Conditions*	*Products**
1	Pyridine–HMDS-TMCS (10:2:1)	1 h at room temp.	1, 2, 3, u
2	Pyridine–BSA (1:1)	1 h at room temp.	1, 2, 3, 4, u
3	Pyridine–acetonitrile–BSA (100:25:75)	1 h at room temp.	1, 2, 3, 4, u
4	Pyridine–BSTFA (1:1)	1 h at room temp.	1, 2, 3, 4, u
5	Pyridine–TMSI (1:1)	1 h at room temp.	1, 2, 3, 4, u
6	PIP–TMSI (1:1)	2 months at room temp.	2, 3, 4, 6
7	PIP–BSTFA (1:1)	2 months at room temp.	2, 3, 4, 6

* u = unknown.

Reagent 1 (pyridine–HMDS–TMCS, 10:2:1), the mildest reagent used, gave TMS-9-enol-PGE_2-Me (2), TMS-PGA_2-Me (3), TMS-PGE_2-Me (1) and an unknown peak (u) with a peak-height ratio of 8:3:4:2.5 (structures are shown in Fig. 1). Reagent 2 (pyridine–BSA, 1:1), reagent 3 (pyridine–acetonitrile–BSA, 100:25:75) and reagent 4 (pyridine–BSTFA, 1:1) showed almost the same peak pattern, consisting of 2, 3, 1, 4 and u with a peak-height ratio of 1:8:3:1:2.5. Reagent 5 (pyridine-TMSI, 1:1) gave a large amount of 2 and several small peaks. The peak-height ratio of 2, 3, 1, 4 and u was 9:3:1:1.2:1. In the reaction using piperidine as the catalyst, as described above, TMSI–PIP (reagent 6) gave small amounts of 2 and 3 and large amounts of 6 and 4, with a peak-height ratio of 0.2:0.2:1.4:2.2. Reagent 7 (BSTFA–PIP) gave overwhelmingly peaks of 6 and 2, with peaks of 3 and 4 as minor components, the peak-height ratio being 50:20:2:2.

These results suggest that the method of Roselló and co-workers is to be preferred for the determination of PGE by GC–MS as it gives a single peak of TMS-9-enol-PGE-Me. However, it is necessary to make it stable for about at least 2 weeks in order to analyse many samples, or to discriminate between the 9-enol peak and the PGF peak in the gas chromatogram.

Further studies are in progress.

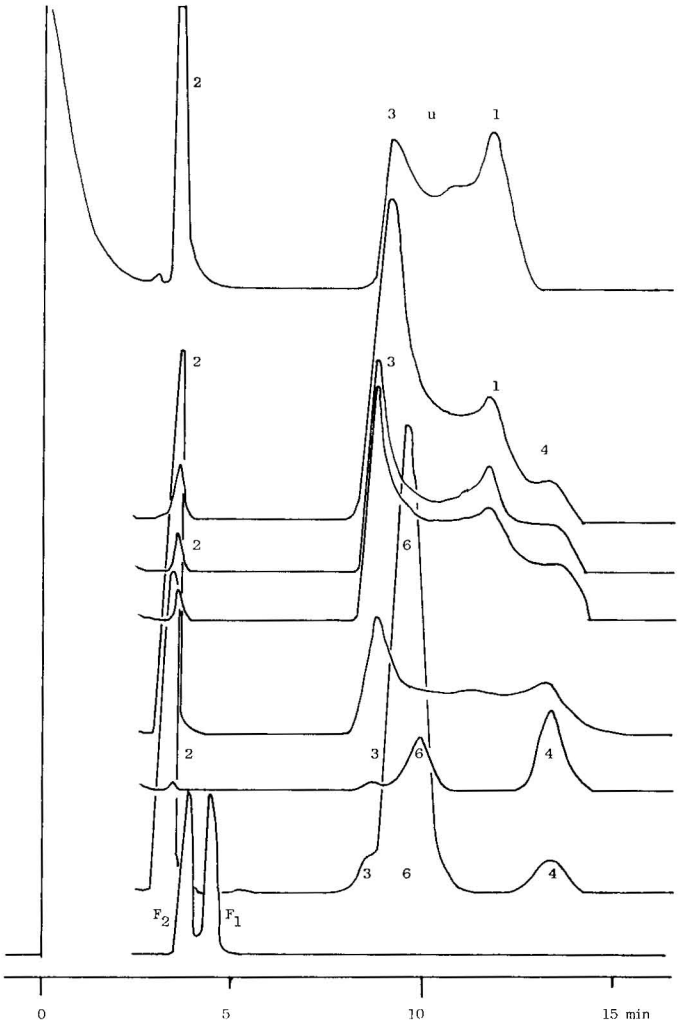

Fig. 5. Gas chromatograms of the reaction products of PGE_2-Me with various trimethylsilylating reagents on an OV-210 column (reagents 1–7, reading downwards).

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SEPARATION OF THE TRYPTIC PEPTIDES FROM REDUCED, ALKYLATED HEN EGG WHITE LYSOZYME BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

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SUMMARY

We describe high-performance liquid chromatographic systems for the separation of the tryptic peptides of reduced, alkylated hen eggwhite lysozyme. The resolved peptides which contained 3–23 amino acid residues were identified by determination of amino acid composition. Gradients of acetonitrile with aqueous ammonium acetate or ammonium chloride were employed to elute from a reversed-phase C_{18} column, monitoring absorbance at 205 nm, 212 nm or 280 nm. Peptides containing S-carboxymethyl-cysteine eluted more rapidly than the corresponding S-ethylsuccinimido peptides.

INTRODUCTION

As part of a series of investigations to determine the folding pathway of reduced lysozyme, an analytical method for tryptic peptides of that protein was required. The proposed application involves separating, estimating, and identifying cysteine-containing peptides in the series of intermediates that arise in the oxidation of the reduced protein. We wanted a method that was rapid, reliable, and sensitive, with high resolving power. For further examination of peptide fractions, it is also desirable to desalt the fractions by sublimation.

At the time these studies were begun, relatively few high-performance liquid chromatographic (HPLC) separations of tryptic peptide mixtures had been achieved[1–3,4]. After unsuccessfully attempting to adapt various published systems to our objective, we began to get encouraging results with a reversed-phase C_{18} column packing and gradient elution with ammonium salt and acetonitrile. Development of the systems was assisted by pilot reversed-phase thin-layer chromatographic (TLC) experiments. Hen egg white lysozyme (HEWL) has 129 amino acids and should theoretically yield 14 tryptic peptides as well as free lysine, arginine and leucine. In this paper we describe HPLC gradient systems which resolve most of the tryptic peptides of reduced alkylated lysozyme. The identities of the peptides have been established by amino acid analysis. While several recent publications have demonstrated the ability of reversed-phase systems to separate tryptic peptides[1,3–8] only a

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few studies have determined the amino acid composition of tryptic peptides collected from HPLC[2,3,8–13]. We believe that our experience in developing successful systems will be useful to other investigators.

MATERIALS AND METHODS

General methods

Sephadex G-25 medium and SP-Sephadex C-25-120 were obtained from Pharmacia (Uppsala, Sweden). Amberlite AG 1-X2, 200–400 mesh, was from Bio Rad Labs. (Richmond, CA, U.S.A.).

Effluents from Sephadex and ion-exchange columns were monitored for absorbance at 230 nm on a Cecil Model CE 272 ultraviolet spectrophotometer (Cecil, Cambridge, Great Britain). Subsequently the fractions were read manually at 280 nm on a Zeiss Model MAQ III spectrophotometer (Oberkochen, Württenberg, G.F.R.).

Column fractions were collected on an LKB 7000 Ultrarac fraction collector (LKB, Stockholm, Sweden).

For amino acid analysis degassed samples were hydrolyzed at 110°C with either 6 *M* hydrochloric acid or 4 *M* methanesulfonic acid (Pierce, Rockford, IL, U.S.A.) for 24–72 h. A Beckman Model 119C amino acid analyzer (Beckman, Palo Alto, CA, U.S.A.) equipped with a single column of Beckman Type AA-20 resin was employed.

HPLC separations were performed on a Varian Model 5000 liquid chromatograph (Varian, Palo Alto, CA, U.S.A.) equipped with a Vari-chrom variable-wavelength detector and a Fischer Recordall Series 5000 dual-pen recorder (Fisher Scientific, Pittsburgh, PA, U.S.A.). A Varian reversed-phase Micropak MCH-10 column (30 cm × 4 mm I.D.) was employed with a guard column of Vydac RP resin. The reagents were analytical grade and the ammonium salt buffers were prefiltered through a Millipore Type HA 0.45-μm filter. Water was purified on a Milli-Q system containing the following cartridges: one activated carbon, two mixed-bed deionization, and one 0.22-μm membrane filter (Millipore, Bedford, MA, U.S.A.). HPLC-grade acetonitrile was obtained from Burdick & Jackson Labs. (Muskegon, MI, U.S.A.).

Preparation of tryptic digests of reduced, S-alkylated HEWL

The four disulfides of HEWL (Miles Labs., South Africa) were reduced with dithiothreitol at pH 8.6 in the presence of urea and EDTA as previously described[14]. The reduced lysozyme was S-alkylated with either N-ethylmaleimide[15] or iodoacetic acid[16] to form respectively S-ethylsuccinimido lysozyme (ES_8LZM) and S-carboxymethyl lysozyme (CM_8LZM). Complete S-alkylation was confirmed by amino acid analysis and by a negative reaction with 5,5′-dithiobis-2-nitrobenzoic acid (Ellman's reagent).

Tryptic digestion of the S-alkylated lysozyme preparations was performed essentially by the method of Canfield[17]. Samples of 10–100 mg were dissolved at final concentrations of 0.05–1.0% in 0.10 *M* acetic acid, 10^{-3} *M* in calcium chloride. The pH was adjusted to 7 with dilute ammonium hydroxide. Diphenylcarbamyl chloride (DPCC)-treated trypsin (Sigma, St. Louis, MO, U.S.A.) was dissolved in 0.10 *M* acetic acid to a concentration of 1 mg/ml. Two separate additions of trypsin were

made to total 2–3 % of the weight of the substrate. The pH was maintained and the digestion carried out for 18–24 h. The digestion was stopped by lyophilization.

Preliminary fractionation of the CM_8LZM and ES_8LZM tryptic peptides on a Sephadex G-25 medium column

The lyophilized tryptic digests from 10–24 mg of CM_8LZM or ES_8LZM were dissolved in approximately 3 ml of 0.10 *M* acetic acid and applied to a Sephadex G-25 medium column (110 × 2.5 cm I.D.). The peptides were eluted with 0.10 *M* acetic acid. Fractions (2 min) containing 5–6 ml were collected. Fractions were pooled, lyophilized and reconstituted in 3–6 ml of 0.10 *M* acetic acid for HPLC chromatography.

Preliminary anion-exchange chromatography of the CM_8LZM and ES_8LZM tryptic peptides

The lyophilized tryptic digests from 50 mg of CM_8LZM or ES_8LZM were dissolved in 5–10 ml of 0.10 *M* acetic acid and applied to a SP-Sephadex (NH_4^+) column (46 × 1.5 cm). The peptides were eluted first with a three-chamber gradient of 500 ml each 0.040 *M* ammonium acetate (pH 3.80), 0.060 *M* ammonium acetate (pH 4.10) and 0.080 *M* ammonium acetate (pH 4.50) and then with a two-chamber gradient of 500 ml each 0.080 *M* ammonium acetate (pH 4.50) and 0.15 *M* ammonium acetate (pH 5.50). The column was washed finally with a 0.15 *M* ammonium acetate buffer made 4 *M* in urea and 0.10 *M* in sodium chloride. Fractions (7 min) of approximately 8.5 ml were collected. UV-Absorbing fractions (10 μl) were examined by HPLC. Those giving no peak on HPLC or a single HPLC peak were hydrolyzed (1 ml) and analyzed for amino acid composition. HPLC chromatography itself was used to resolve multipeak fractions.

Preliminary cation-exchange chromatography of the CM_8LZM and ES_8LZM tryptic peptides

The lyophilized tryptic peptides from 50 mg CM_8LZM or ES_8LZM were dissolved in 3–5 ml of 0.01 *M* ammonium acetate and applied to an Amberlite AG 1-X2 (CH_3COO^-) column (46 × 1.5 cm I.D.). The peptides were eluted with a three-chamber gradient of 500 ml each; 0.050 *M* acetic acid, 0.50 *M* acetic acid and 2.00 *M* acetic acid. The column was washed finally with an additional 500 ml of 2 *M* acetic acid. Fractions (3 min) of approximately 4.2 ml were collected. UV-Absorbing fractions (10 μl) were examined directly by HPLC and those showing no peak or a single 205 nm absorbance peak were hydrolyzed (1 ml) and analyzed for amino acid composition. Multipeak fractions were resolved by HPLC chromatography.

HPLC gradient systems for mapping tryptic peptides

Separation of the tryptic peptides on HPLC was first achieved using either 0.10 *M* ammonium acetate or 0.10 *M* ammonium chloride, pH 4.1 (reservoir A) and acetonitrile (reservoir B) in the following gradient program: 0–10 min, 5–22 % B; 10–12 min, 22–24 % B; 12–14 min, 24 % B; 14–19 min, 24–28 % B; 19–25 min, 28–36 % B (system I). The conditions were: flow-rate, 1.5 ml/min; chart speed, 1 cm/min; UV detection, 280 nm for the ammonium acetate system and 205 nm for the ammonium chloride system; range, 0.05 a.u.f.s. for ammonium acetate and 0.10 units for ammonium chloride.

A modified system used 0.010 *M* ammonium chloride or 0.010 *M* ammonium acetate, pH 4.2 (reservoir A) and acetonitrile (reservoir B) in the following program: 0–20 min, 5–25% B; 20–30 min, 25–40% B (system II). Other conditions were the same as for system I, except that for ammonium acetate 212 nm detection could be used with a full scale range of 0.2 absorbance unit.

When HPLC fractions were collected, ammonium acetate buffers were used. For System I, peptides which did not absorb at 280 nm were collected by time based on their elution profile in the chloride system. For collection the back-pressure restrictor and exit line from the Vari-chrom detector were replaced with tubing having a total volume of 400 μl. Injections of the different peptide preparations were made and the peaks collected manually. For the Sephadex G-25 column pool samples, peaks from six to ten 10 μl injections were combined, lyophilized, hydrolyzed and the entire sample applied to the amino acid analyzer (0–5 nmol scale). For fractions from the ion-exchange columns, peaks from a single injection of 2–10 μl were collected for amino acid analysis.

RESULTS AND DISCUSSION

The preliminary fractionation of a tryptic digest of ES_8LZM using Sephadex G-25 is shown in Fig. 1. The fractions were pooled as shown, following Anderson and Wetlaufer[18]. Tryptic digests of CM_8LZM gave similar elution profiles to those of ES_8LZM on Sephadex G-25. Pool D had an elution volume equal to the total column volume, suggesting a hydrophobic composition. Both pools C and D have higher 280 nm absorbance than pools A and B.

The HPLC gradient system using acetonitrile and 0.10 *M* ammonium chloride was developed to resolve the peptides in these four pools. Fig. 2 shows the elution profiles of these pools and Tables I and II give the amino acid analyses of the collected

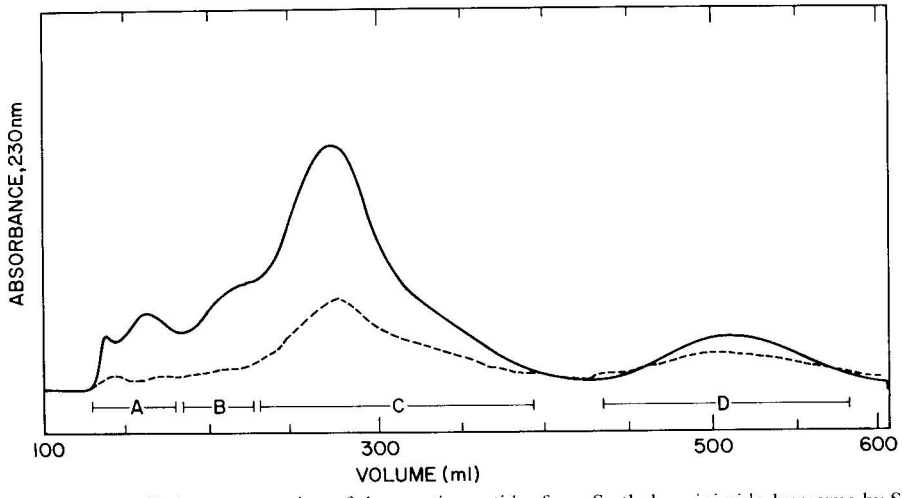

Fig. 1. The preliminary separation of the tryptic peptides from S-ethylsuccinimido lysozyme by Sephadex G-25 chromatography. The column was eluted with 0.10 *M* acetic acid and four peptide pools A, B, C and D were made by combining fractions as indicated. ——, Absorbance at 230 nm; - - -, absorbance at 280 nm.

peaks. Peptide assignments were made considering the known sequence of HEWL[17]. The three major peaks from pool B of the ES_8LZM digest (Fig. 2A) were identified as peptides Phe^{34}–Arg^{45} and a doublet of ES-Cys^{6}–Lys^{13} and Asn^{46}–Arg^{61}. The following peptides were found in pool C: Thr^{69}–Arg^{73}, His^{15}–Arg^{21}, Gly^{117}–Arg^{125}, Ile^{98}–Arg^{112} and Gly^{22}–Lys^{33}. The latter two peptides elute as one peak with ammonium chloride, but separate well with ammonium acetate (Fig. 2B). Pool D contained essentially pure Trp^{62}–Arg^{68} (Fig. 2C). The order of elution of the tryptic peptides from the Sephadex G-25 column is consistent with conventional expectations. The pool B peptides have eight, twelve and sixteen amino acid residues, and contain no tryptophan. The three large peptides found in pool C have nine, twelve, and fifteen amino acid residues, including one or two tryptophans. Trp^{62}–Arg^{68} has seven amino acids, two of which are tryptophan, making plausible its retention on Sephadex G-25.

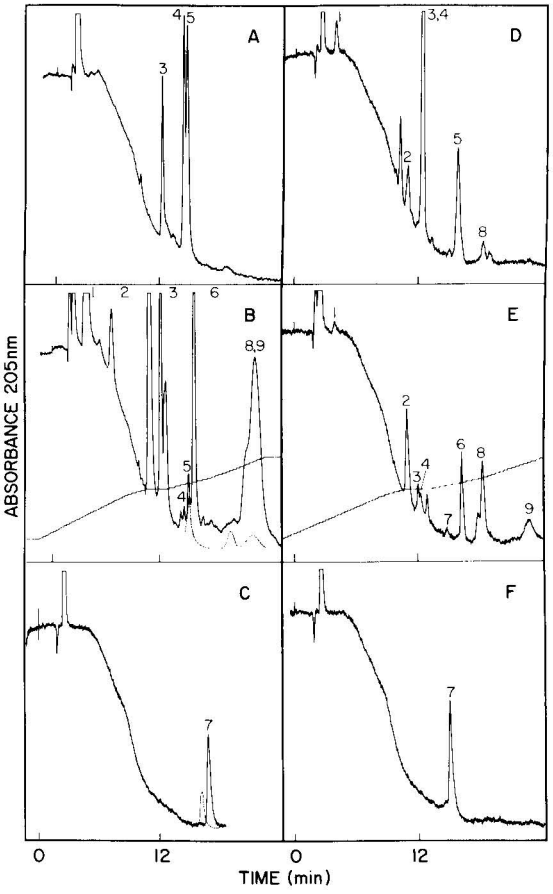

Fig. 2. HPLC separation of the lysozyme tryptic peptide pools collected from Sephadex G-25 medium chromatography. HPLC gradient system I with ammonium chloride was used as described in the text. (A), Pool B from ES_8LZM digest; (B), pool C from ES_8LZM digest; (C), pool D from ES_8LZM digest; (D), pool B from CM_8LZM digest; (E), pool C from CM_8LZM digest and (F), pool D from CM_8LZM digest. The numbers above the peaks refer to the peptides identified in the tables. The gradient pen is offset 1.5 min to the left of the actual elution time. The dashed curves show the tryptophan peptides with ammonium acetate in the eluent.

TABLE I

THE AMINO ACID COMPOSITION OF TRYPTIC PEPTIDES OF N-ETHYLMALEIMIDE BLOCKED HEN EGG WHITE LYSOZYME SEPARATED BY HPLC

The data are given in residues/mole. The numbers in parentheses refer to the number of residues expected from the known sequence of lysozyme. Residue numbers are given after hydrolysis in 4 *M* methane sulfonic acid.

Amino acids	*HPLC peak No. (Fig. 2)*								
	1	*2*	*3*	*4 and 5*	*6*	*7*	*8*	*9*	*Between 2 and 3 10*
	Peptide(s) identified								
	Thr^{69}–Arg^{73}	*His^{15}–Arg^{21}*	*Phe^{34}–Arg^{45}*	*Cys^{6}–Lys^{13} and Asn^{46}–Arg^{61}*	*Gly^{117}–Arg^{125}*	*Trp^{62}–Arg^{68}*	*Gly^{22}–Lys^{33}*	*$Ileu^{98}$–Arg^{112}*	*Lys^{1}–Arg^{5}*
S-Ethyl-succinimido cysteine				0.4 (1)		1.4 (1)	0.6 (1)	1.6	
Aspartic acid		2.2 (2)	3.3 (3)	3.6 (4)	1.3 (1)	2.3 (2)	1.3 (1)	3.2 (3)	0.2
Threonine	0.9 (1)		1.9 (2)	2.1 (2)	1.1 (1)				
Serine	0.9 (1)	0.2	1.1 (1)	2.0 (2)			0.7 (1)	1.3 (1)	
Glutamic acid			2.5 (2)	2.4 (2)	1.2 (1)		0.1	0.4	0.3
Proline	1.0 (1)								
Glycine	1.2 (1)	1.3 (1)	1.0	2.5 (2)	1.4 (1)	1.3 (1)	2.7 (2)	2.8 (2)	0.7 (1)
Alanine			1.9 (1)	2.8 (3)	1.2 (1)		2.5 (2)	2.3 (2)	
Valine					0.8 (1)		1.5 (1)	2.0 (2)	0.5 (1)
Methionine				0.8 (1)				0.4 (1)	
Isoleucine				1.7 (2)	0.9 (1)		0.4	0.6 (1)	
Leucine		1.1 (1)		2.1 (2)			1.1 (1)		0.2
Tyrosine		0.7 (1)		0.8 (1)			0.7 (1)		0.4
Phenylalanine			2.5 (2)						0.7 (1)
Histidine		1.0 (1)	0.2						
Lysine			0.2	0.9 (1)			1.0 (1)		0.6 (1)
Arginine	1.1 (1)	1.0 (1)	1.0 (1)	1.0 (1)	1.0 (1)	1.0 (1)	0.2	1.0 (1)	1.0 (1)
*Tryptophan					0.9 (1)	1.6 (2)	+ (1)	+ (2)	

* Tryptophan is indicated present after 6 *M* HCl hydrolysis by +.

TABLE II

THE AMINO ACID COMPOSITION OF TRYPTIC PEPTIDES OF IODOACETIC ACID BLOCKED HEN EGG WHITE LYSOZYME SEPARATED BY HPLC

The data are given in residues/mole. The numbers in parentheses refer to the number of residues expected from the known sequence of lysozyme.

Amino acids	*HPLC peak No. (Fig. 2)*							
	1	*2*	*3 and 4*	*7*	*5*	*6*	*8*	*9*
	Peptide(s) identified							
	Thr^{69}–Arg^{73}	*His^{15}–Arg^{21}*	*Cys^{6}–Lys^{13} and Phe^{34}–Arg^{45}*	*Try^{62}–Arg^{68}*	*Asn^{46}–Arg^{64}*	*Gly^{117}–Arg^{125}*	*Gly^{22}–Lys^{33}*	*$Ileu^{98}$–Arg^{112}*
Carboxymethylcysteine			0.8 (1)	1.3 (1)			1.1 (1)	
Aspartic		2.0 (2)	2.5 (3)	2.4 (2)	4.0 (4)	1.5 (1)	2.1 (1)	2.6 (3)
Threonine	1.1 (1)		1.9 (2)		2.0 (2)	1.2 (1)		
Serine	1.1 (1)	0.8	1.0 (1)	0.1	2.0 (2)	1.4	1.5 (1)	1.1 (1)
Glutamic acid		0.8	2.8 (3)		1.6 (1)	1.6 (1)		0.6
Proline	1.1 (1)						0.3	
Glycine	1.2 (1)	2.1 (1)	0.6	1.0 (1)	2.7 (2)	2.4 (1)	2.5 (2)	2.6 (2)
Alanine		0.5	4.5 (4)		0.9	1.5 (1)	2.1 (2)	2.0 (2)
Valine						1.1 (1)	1.3 (1)	1.9 (2)
Methionine			0.9 (1)					1.5 (1)
Isoleucine					3.0 (2)	1.0 (1)		1.7 (1)
Leucine		1.2 (1)	1.6 (1)		2.2 (1)	0.2	1.1 (1)	
Tyrosine		1.0 (1)			1.9 (1)		1.0 (1)	
Phenylalanine			1.4 (2)					
Histidine		0.9 (1)						
Lysine	0.1		1.2 (1)				1.0 (1)	
Arginine	1.0 (1)	1.0 (1)	1.0 (1)	1.0 (1)	1.0 (1)	1.0 (1)		0.7 (1)
Tryptophan				1.5 (2)		0.7 (1)	1.1 (1)	1.3 (2)

All the tryptophan-containing peptides elute late in the HPLC gradient, consistent with their relatively higher hydrophobicity. In some chromatograms of the pool C peptides a peak is seen between His^{15}–Arg^{21} and Phe^{34}-Arg^{45}; this has been identified as Lys^{1}–Arg^{5}, showing that trypsin did not completely cleave the Lys^{1}–Val^{2} bond. This peptide has been found previously in tryptic digests of lysozymes[17,19].

For comparison, the HPLC maps of the corresponding pools from a CM_8LZM digest are shown in Fig. 2D–F. Pool B contains Asn^{46}–Arg^{61} and a single peak of both Phe^{34}–Arg^{45} and CM-Cys^{6}–Lys^{13}. Pool C had the same peptide content as the corresponding ES_8LZM pool; likewise pool D was Trp^{62}–Arg^{68} (Table II). The three S-carboxymethylated cysteinyl peptides (Cys^{6}–Lys^{13}, Gly^{22}-Lys^{33} and Try^{62}–Arg^{68}) elute more quickly than the corresponding S-ethylsuccinimido cysteinyl peptides. The amino acid analyses of the pool B peptides (Tables I and II) show the CM-Cys^{6}–Lys^{13} peptide eluting with Phe^{34}–Arg^{45}, the ES-Cys^{6}–Lys^{13} peptide elutes later, very close to Asn^{46}–Arg^{61}. The second half of this doublet (peaks 4 and 5) elutes in

TABLE III

THE AMINO ACID COMPOSITION OF OTHER TRYPTIC PEPTIDES OF HEN EGG WHITE LYSOZYME SEPARATED BY ION-EXCHANGE CHROMATOGRAPHY

The data are given in residues/mole. The numbers in parentheses refer to the number of residues expected from the known sequence of lysozyme.

Amino acids	*HPLC peak No. (Figs. 2 and 3)*					
	11	*11*	*12*	*13*	*10*	*3*
	Peptide identified					
	Gly^{126} Arg^{128} (CM)	*Gly^{126} Arg^{128} (ES)*	*Gly^{126} Leu^{129} (CM)*	*Val^{2} - Arg^{5}*	*Lys^{1} Arg^{5}*	*Phe^{34} - Arg^{45}*
S-Carboxymethyl-cysteine	0.9 (1)		0.7 (1)			
S-Ethylsuccinimido-cysteine		0.6 (1)				
Aspartic acid	0.2	0.2				2.7 (3)
Threonine						1.9 (2)
Serine						1.3 (1)
Glutamic acid	0.1					2.0 (2)
Proline						
Glycine	1.0 (1)	1.2 (1)	1.2 (1)	1.2 (1)	0.9 (1)	0.5
Alanine		0.2	0.3	0.4	0.2	1.4 (1)
Valine				0.7 (1)	1.3 (1)	
Methionine						
Isoleucine						
Leucine	0.1	0.2	1.1 (1)		1.0 (1)	
Tyrosine						
Phenylalanine				0.7 (1)	0.8 (1)	1.6 (2)
Histidine						
Lysine		0.2				
Arginine	1.0 (1)	1.0 (1)	1.0 (1)	1.0 (1)	0.9 (1)	1.0 (1)
Tryptophan						

the same position as Asn^{46}–Arg^{61} in the carboxymethylated pool, allowing the identity and positions of the three peptides to be deduced. In pool C the carboxymethylated form of Gly^{22}–Lys^{33} is readily separated from Ile^{98}–Arg^{112} in either chloride or acetate buffer, unlike the S-ethylsuccinimido peptide (Fig. 2B and E). The S-ethylsuccinimido derivative of Trp^{62}–Arg^{68} is well separated from the other peptides while the carboxymethylated form elutes close to Asn^{46}–Arg^{61} and Gly^{117}–Arg^{125} (Fig. 2C–F). Fortunately, this overlap can be avoided by preliminary fractionation on Sephadex G-25.Therefore, by suitable choice of alkylating agent, improved separation and isolation of peptides is achieved. The longer retention times of the ES peptides suggests that the S-ethylsuccinimido group is less polar than the carboxymethyl group.

Preliminary fractionation of HEWL tryptic peptides by ion-exchange chromatography provided independent confirmation of the peak assignments made in Tables I and II. The identification of the doublet peaks 4 and 5 of ES_8LZM digest pool B (Fig. 2A) as ES-Cys^6–Lys^{13} and Asn^{46}–Arg^{61} (Table I) was confirmed by the injec-

4	*14*	*14*	*15*	*15*	*16*	*17*
CM-Cys^6–Lys^{13}	*CM-Cys^6–Arg^{14}*	*ES-Cys^6–Arg^{14}*	*Asn^{74}–Lys^{96} (CM_3)*	*Asn^{74}–Lys^{96} (ES_3)*	*Val^{109}–Arg^{112}*	*Gly^{117}–Trp^{123}*
1.1 (1)	0.9 (1)		3.1 (3)			
		0.7 (1)		1.5 (3)		
	0.3	0.3	3.6 (4)	4.2 (4)	0.2	1.3 (1)
		0.1	1.0 (1)	1.3 (1)		0.9 (1)
		0.2	3.9 (4)	4.4 (4)		0.4
1.1 (1)	1.2 (1)	1.2 (1)				0.9 (1)
			1.0 (1)	1.2 (1)		
	0.3	0.4	0.4	0.4	0.2	1.3 (1)
3.0 (3)	3.4 (3)	2.7 (3)	3.1 (3)	3.4 (3)	1.1 (1)	1.4 (1)
			0.9 (1)	1.3 (1)	0.8 (1)	1.0 (1)
1.0 (1)	0.8 (1)	1.0 (1)				
			1.9 (2)	1.8 (2)		
1.1 (1)	0.7 (1)	1.1 (1)	2.8 (3)	2.6 (3)		
0.5						
1.0 (1)	1.0 (1)	0.8 (1)	1.0 (1)	1.0 (1)		
	1.0 (1)	0.9 (1)			1.0 (1)	
					0.9 (1)	1.0 (1)

tions of SP-Sephadex pool 139–154 (ES-Cys^{6}–Lys^{13}) and pool 42–46 (Asn^{46}–Arg^{61}) which gave peaks 4 and 5 respectively. Similarly, the identification of a peak of CM_8LZM digest pool B (Fig. 2D) as containing both CM-Cys^{6}–Lys^{13} and Phe^{34}–Arg^{45} was verified by both amino acid analyses of Amberlite AG 1-X2 fractions 21–40 and 99–106 (Table III) and co-chromatography in HPLC System I.

The preliminary fractionation of HEWL tryptic peptides by ion-exchange chromatography also provided additional HPLC peak assignments (Table III). Some of these are peptides resulting from incomplete trypsin cleavage where there are adjacent cleavage sites. These peptides have been reported previously in the literature[17,19,20]. Two peptides Gly^{117}–Trp^{123} and Val^{109}–Arg^{112} require cleavages of Trp–Val and Ile–Trp, bonds not expected to be cleaved by DPCC-treated trypsin.

The elution profiles found for HEWL tryptic peptides from the ion-exchange columns were similar to those found for cation-exchange chromatography by Canfield[17] and Jolles *et al.*[19] and for anion-exchange chromatography by Anderson and Wetlaufer[18] and Fujio *et al.*[21]. The omission of pyridine from the buffer systems somewhat altered the order of elution but did not appreciably affect the yields of peptides. The absence of pyridine did allow the direct monitoring of peptide elution by absorbance at 230 nm; peptides of three or more residues were easily detected.

While HPLC System I has been useful in separating the peptides containing 5–16 amino acid residues, it suffers two limitations. First, the peptide Asn^{74}–Lys^{96}, known to be in pool A, is not eluted. Although it contains three cysteinyl residues neither alkylated form could be consistently eluted. Second, the free amino acids and smaller peptides were not separated. An experiment was performed using the material eluting early from the HPLC run of CM-pool C to demonstrate their presence as a group. Sequentially timed collections were made between 1.5 and 3.5 min and amino acid analyses on each obtained. In this way the presence of Cys^{115}–Lys^{116}, free arginine, free lysine, free leucine, Asp^{113}–Arg^{114}, Gly^{126}–Cys^{127}–Arg^{128} and Val^{2}–Phe^{3}–Gly^{4}–Arg^{5} was demonstrated. Thus the remainder of the set of theoretical tryptic peptides from HEWL were accounted for.

The Amberlite AG 1-X2 chromatography gave better resolution for the CM_8LZM digest and the SP-Sephadex chromatography was more effective for the ES_8LZM digest. CM-cysteine is more acidic than ES-cysteine; therefore, the CM_8LZM digest has more neutral and acidic peptides while the ES_8LZM digest has more basic peptides. Peptides separated by ion exchange are often examined and further purified by paper chromatography or TLC and/or electrophoresis prior to amino acid analysis. HPLC chromatography proved to be an excellent alternative for this purpose.

HPLC Gradient system II proved to give an excellent overall resolution of the tryptic peptides of reduced, alkylated HEWL. The results of injection of unfractionated tryptic digests are shown in Fig. 3. The identification of the peaks (see Tables I, II and III) was deduced by injections of Sephadex G-25 pools and ion-exchange column fractions, and by comparison of the absorbances at 280 nm and 205 nm to further verify the tryptophan-containing peptides. The Phe^{34}–Arg^{45} and CM-Cys^{6}–Lys^{13} peaks, which eluted together in system I, were well-resolved in system II. Peptides Trp^{62}–Arg^{68} (CM), Asn^{46}–Arg^{61}, and Gly^{117}–Arg^{125} which clustered in the first system were now well separated. Ile^{98}–Arg^{112} and Gly^{22}–Arg^{33} (ES) separate in either chloride or acetate, using system II. The largest lysozyme peptide Asn^{74}–Lys^{96},

blocked with either alkylating agent, did elute in system II, although the tricarboxymethylated derivative coelutes with Gly^{22}–Lys^{33} (CM). The two tetrapeptides (Val^{2}–Arg^{5} and Gly^{126}–Leu^{129}) and the tripeptide (Gly^{126}–Arg^{128}) were resolved and identified by amino acid analysis (Table III). Work to locate and identify the dipeptides is still in process. Collection and identification of these small peptides should proceed easily since direct detection at 212 nm is possible with the low acetate concentration of system II.

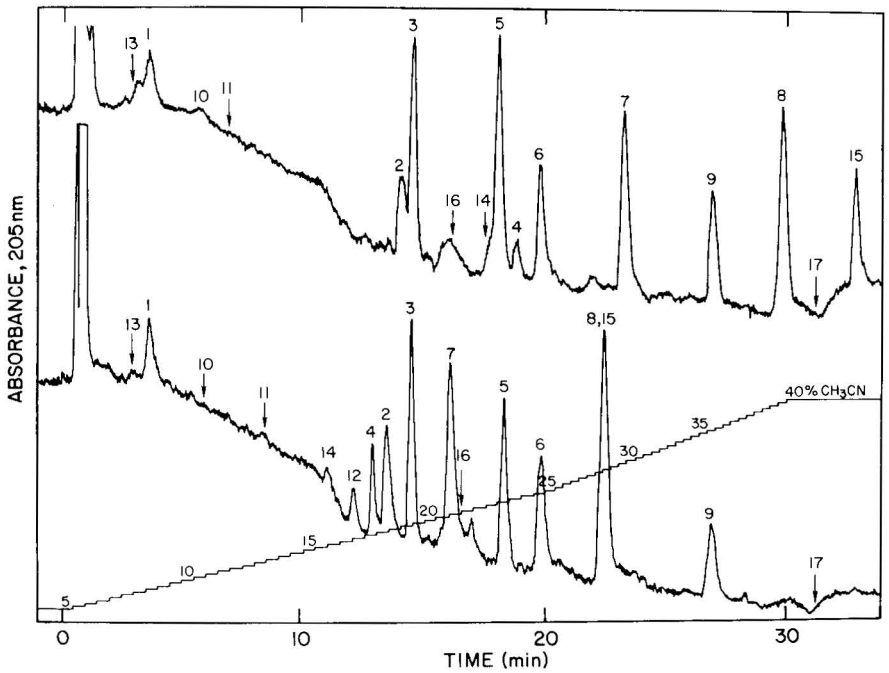

Fig. 3. HPLC separation of the unfractioned tryptic peptides of HEWL, using HPLC gradient system II with ammonium chloride as described in the text. The top tracing is the separation of ES_8LZM digest and the bottom that of CM_8LZM digest. In each case a digest of 1.3 μg or 90 pmol lysozyme was injected. The numbers above the peaks refer to the peptides identified in the tables. The gradient pen is 1.5 min to the right of the actual elution time.

A general comment as to the stability of the Varian MCH-10 column, packed with uncapped C_{18} material, seems warranted. Our column has been in constant use for 2 years. It has been used with other salts and with methanol as an alternative organic solvent. The column is stored in 100% acetonitrile after thoroughly washing the salt out of the system. The Sephadex column pool C peptides have been used as an internal standard. Their elution pattern has been constant once the column has been re-equilibrated. We have found that as much as four hours is necessary for aqueous acetonitrile equilibration after methanol use. The guard column resin is changed every 6 to 8 weeks.

The purposes of this investigation were twofold. First to develop a system(s) suitable for the separation of a group of tryptic peptides from lysozyme which might be useful for other protein digests. Second to modify cysteine residues within these

peptides with two different kinds of alkylating agents to determine which derivative was more suitable for resolution on a reversed-phase column. The use of ammonium salts and an acetonitrile gradient has resulted in the separation of a large number of peptides containing 3–23 amino acid residues. While our system was being developed Coy[9] published a figure showing the separation of the tryptic peptides of β_h-endorphin on LiChrosorb RP-18 with a linear gradient of 10–50% isopropanol–ammonium acetate, pH 4. The author states that substitution of acetonitrile for isopropanol improved resolution. This system, which is similar to ours, was used to separate only five peptides, while in the present study as many as seventeen peptides are separated. Two HPLC separations on μBondapak C_{18} of tryptic peptides from hemoglobin variants[11,12] have recently been published. They also employ ammonium acetate (pH 6.07 and pH 5.7) and acetonitrile gradients. Our substitution of ammonium chloride for ammonium acetate in order to detect peaks at 205 nm showed the two salts to be comparable eluants. We conclude therefore that ammonium salt (pH 4–6 range)–acetonitrile systems are generally effective for peptide mapping. The sensitivity found here for peptide detection by 205 nm absorbance appears to be about the same as that reported by Rubinstein and co-workers[2,8] employing fluorescence detection. Our studies also show that S-ethylsuccinimido- and S-carboxymethyl-cysteinyl peptides are equally well resolved on reversed-phase columns but their different retention times can be useful in improving the overall separation of a group of peptides. It seems likely that our systems will be applicable to other peptide mixtures.

ACKNOWLEDGEMENT

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Note

Direct desorption of traps for capillary column gas chromatography

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A simple and inexpensive attachment has been described by Peterson *et al.*[1] for the direct sampling of sorption traps used to collect volatile organic compounds. However, I found that their thermal focussing technique of lowering the column temperature below ambient, caused drastic loss of resolution with Carbowax 20M support-coated open tubular (SCOT) columns. This note describes modifications which enable the attachment to be used with any type of column and allow conventional syringe injection without detaching the column and removing the injector insert.

EXPERIMENTAL

Construction

The dimensions given in Fig. 1 are based on a Varian 2700 gas chromatograph fitted with 6.3-mm injectors. The equilibration tube was made from cold-drawn 6.3 mm O.D. stairless-steel tubing. The coupling (N) to the inlet of the injector and the O-ring seal assembly (K) were machined from 12.7-mm brass rod. Both fittings were silver soldered on to the stainless-steel tube. Alternatively the O-ring seal assembly may be made from a 1/8-in. Gould Eastman–Imperial compression fitting. (Needs to be partially drilled out with a 6.3-mm drill so that the O-ring is retained.) The seal (O) between the coupling N and the injection port was made from a low-bleed septum bored out with a cork borer of appropriate size.

The transfer line (I) consisting of 1.6 mm O.D. glass-lined stainless-steel tubing (Scientific Glass Engineering, Ringwood, Australia) was attached to the length of 6.3 mm O.D. stainless-steel tubing (G) by crimping the larger tubing and silver soldering. The PTFE seal (F) makes a tight fit with the 6.3 mm O.D. tubing (G) and the transfer line. The internal diameter of the seat (D) is such that the stainless-steel trap could slide into it without undue force. The transfer line (I) was terminated 3 mm from the seat (D). The syringe injection liner, when it is used, fits into this recess (E).

The traps (J) were made from 85 mm of cold-drawn 3.3 mm O.D. × 2.5 mm I.D. stainless-steel tubing (Fig. 1). One end was tapped so that the plunger (P) could be screwed into it. A carrier gas inlet hole (1.0 mm) was drilled at 10 mm from the threaded end. The other end was rounded over so that it could slide into the seat (D) without damaging it.

A 16-mm hole was drilled through the lid of the column oven. This hole was

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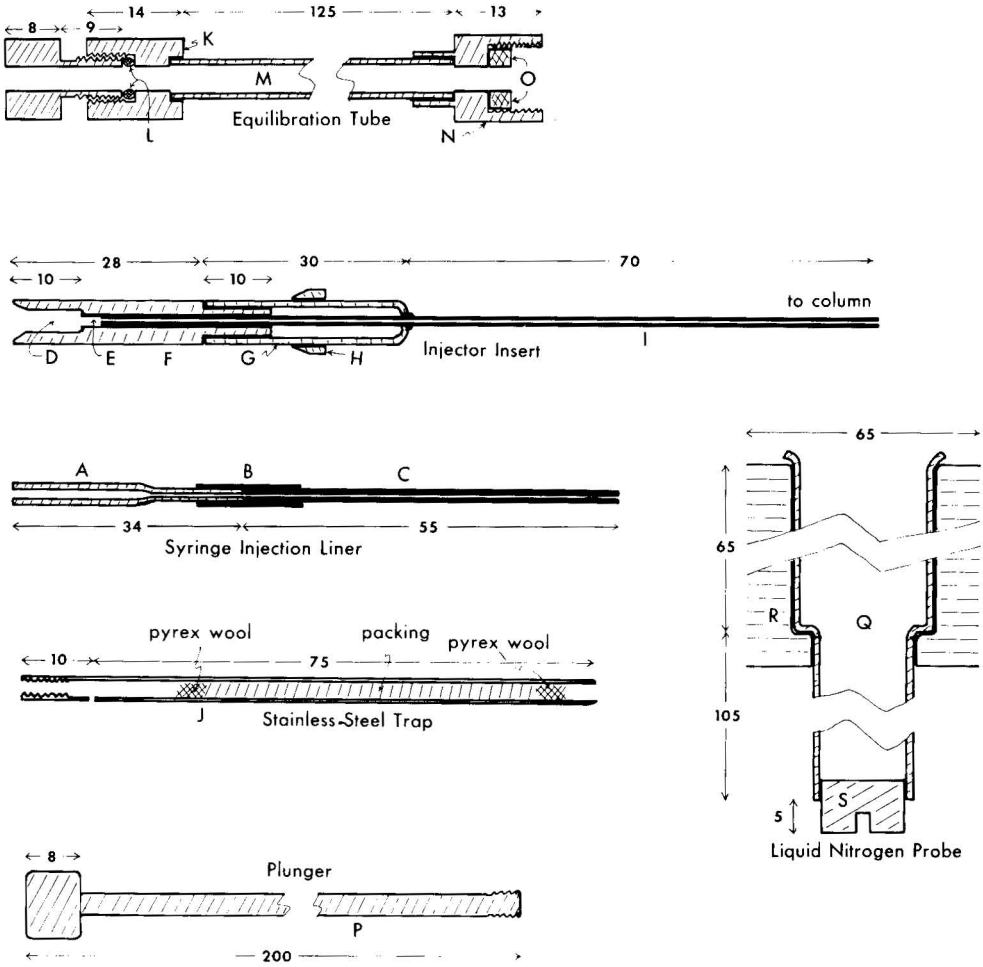

Fig. 1. Direct desorption attachment. All dimensions are in mm. A = 3 mm O.D. Pyrex tubing drawn out as shown; B = heat shrink PTFE tubing; C = 1.6 mm O.D. glass-lined stainless-steel tubing (GLT); D = trap seat (makes sliding seal with trap); E = syringe injection liner seat 3 mm long (makes sliding seal with C); F = PTFE adapter, machined from 6.3 mm rod – makes tight fit with G and I; G = 6.3 mm O.D. stainless-steel tubing; H = Vespel ferrule (Swagelok nut is not shown); I = 1.6 mm O.D. GLT transfer line silver soldered to G (note that it extends up to the seat E); J = trap made from 3.2 mm O.D. cold-drawn stainless-steel tubing threaded (3 mm × 0.5) and drilled (1 mm) through side as shown; K = O-ring seal assembly machined from 12.7-mm brass rod, the I.D. is 3.5 mm; L = 6.3 mm O.D. × 1.6 mm section silicone rubber O-ring; M = 6.3 mm O.D. stainless-steel tubing; N = coupling machined from 12.7-mm brass rod and threaded to match that of the injection port; O = bored-out low-bleed silicone rubber septum; P = plunger made from 3.1-mm stainless-steel rod threaded to take J; Q = liquid nitrogen reservoir made from lengths of 15 mm O.D. and 21 mm O.D. copper tubing silver soldered together; R = expanded polystyrene insulation; S = 12.7 mm copper rod silver soldered to Q. The slot, 3.5 mm deep × 1.8 mm wide, fits over the transfer line I.

positioned so that the liquid nitrogen-cooled probe (Fig. 1) or the heated probe could be quickly applied near the mid-point of the transfer line (I) extending beyond the injector. The heated probe (not shown) consists of 90 mm of 15.9-mm copper rod

terminated at one end with a slot as in S. At the other end 100 mm of 6.3 mm O.D. stainless-steel tubing was silver soldered on for a handle.

Assembly and operation

A short length of 6 mm O.D. × 4 mm I.D. Pyrex tubing was placed inside the injector between the injection port and the injector insert to guide the trap into the PTFE seat (D) during trap insertion. The injector insert was introduced into the injector from the outlet end and secured with a 1/4-in. Swagelok nut. The position of the ferrule (H) was adjusted so that the end of A was approximately 2 mm from the septum when the syringe injection liner is properly seated in E. The liner could be easily removed by withdrawing it through the injection port with the aid of a short length of stiff wire slightly bent at one end, so that it lightly gripped the inside wall of A. I used the plunger from a 10-μl syringe. The column was attached to the end of the transfer line (I) using a zero dead volume union. Next the equilibration tube assembly was attached in place of the septum and septum nut via the coupling N.

The procedure for using the attachment is now described. The test sample was a mixture containing 1 μl of each of ethyl acetate, ethyl butanoate, pentan-3-ol, and pent-1-en-3-ol, in 50 ml of water.

The stainless-steel trap was packed with 100 mg of Chromosorb 105[2] and was conditioned initially at 190°C with nitrogen at 20 ml/min for 48 h. Immediately before use the trap was conditioned for a further 1 h. The non-threaded end of the trap was attached, using a Swagelok reducing union, to 120 mm of 6 mm O.D. Pyrex tubing. A low wattage thermostated tube heater was placed around the Pyrex tubing. A 50-μl aliquot of the aqueous test mixture was deposited into the Pyrex tubing, and flushing into the trap with nitrogen (15 ml/min) over a 20-min period. During this period the temperature of the Pyrex tube was slowly increased to 100°C. The trap containing the sample was flushed with more nitrogen (20 ml/min) at 40°C for 30 min to remove adsorbed water. The plunger was screwed into the trap and the assembly partially inserted through K into the equilibration tube so that the 1 mm hole in the trap was still visible. After 5 min (to allow air to be flushed out of the trap) the assembly was pushed in until the trap was inside the equilibration tube. It was held in this position until the column flow (head pressure) had re-established. The cold probe was lowered through the oven lid so that the transfer line was in the slot of the probe tip (S). Liquid nitrogen was poured into the probe reservoir. The trap was then pushed in until it was seated in the PTFE seat (D). Liquid nitrogen was maintained in the reservoir during the 7-min desorption period. The trap was then withdrawn from the seat but not out of the injector. The cold probe was then quickly exchanged for the heated probe (200°) in order to "inject" the sample.

RESULTS AND DISCUSSION

Fig. 2 shows a chromatogram of the test mixture introduced with the direct desorption attachment in conjunction with a trap packed with Chromosorb 105. The peaks are sharp and symmetrical.

When direct sampling of sorption traps is used with capillary columns some method of sample concentration is essential during the desorption period. I have found the thermal focussing technique in which the column temperature is lowered to ambient or below difficult to control with the Varian 2700 gas chromatograph. It also

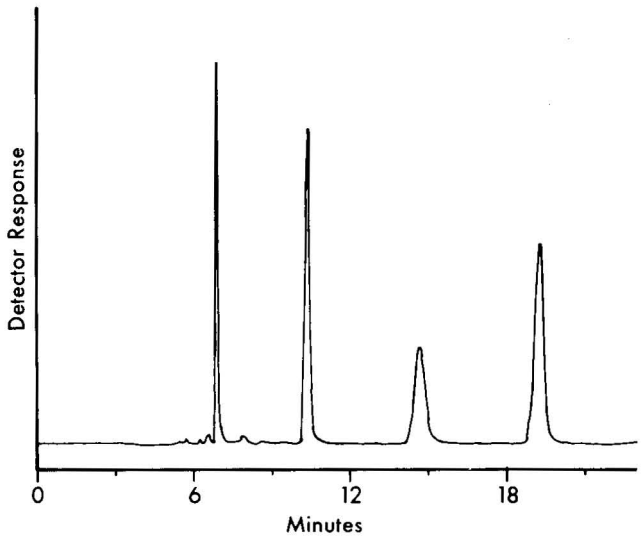

Fig. 2. Typical gas chromatogram obtained with direct desorption attachment. Trap packing: Chromosorb 105. Column: 60 m × 0.5 mm I.D. SCOT coated with Carbowax 20M. Carrier gas: nitrogen at 18 cm/sec. Temperature program: isothermal for 15 min at 50°C then 1°C/min. Temperatures: injector 175°C, detector 150°C. Sample: ethyl acetate, ethyl butanoate, pentan-3-ol, pent-1-en-3-ol (in increasing retention time).

resulted in poor resolution when a Carbowax 20M SCOT column was installed. This is probably due to solidification of the stationary phase. Our cold probe method which is similar to that described by Murray *et al.*[2] overcame these problems.

In over 12 months of intensive use of the direct desorption attachment I have not encountered any problems with accelerated degradation of column performance attributable to the entry of air or water on to the column. However, care has always been taken to lower the column temperature before venting the injector to the atmosphere. Excessive sample or water loading of the trap could cause a blockage of the transfer line during the desorption process but with sample sizes compatible with capillary columns this should not occur. The system has been used at desorption temperatures as high as 230°C without problems. Higher desorption temperatures were avoided by a suitable choice of packing for the trap (*e.g.*, we have used 10% OV-101 on silanised Pyrex wool for farnesol).

This direct sampling accessory makes it very easy to check for homogenity of gas chromatography (GC) peaks by trapping and analysing on liquid phases of different polarity. Samples eluting from the gas chromatograph could be collected directly into a suitably packed trap with little or no cooling in essentially quantitative yields. In cases where a stationary phase unsuitable for GC–mass spectrometry (MS) is necessary to obtain adequate separation, this trapping and re-chromatographing procedure makes it possible for the actual GC–MS analysis to be carried out on a column more compatible with GC–MS with little increase in the amount of sample required.

Syringe injection of samples could be readily accomplished without disconnecting the column to replace the injector insert. Instead the equilibration tube assembly

is removed, the syringe injection liner (Fig. 1) inserted into the seat E, and the injection port sealed with the septum and septum holder in the conventional manner. The ease of changing between direct desorption of traps and syringe injections has proved to be very convenient for checking column performance and for introducing standards, especially for GC–MS analyses.

In the design of the simple and inexpensive desorption device described above, I have given high priority to the ease of using it on an unmodified injector and to the use of dimensions that are compatible with flow-rates associated with capillary columns. I have found the accessory to be invaluable in the GC and GC–MS analyses of a wide range of compounds present at low levels in biological systems.

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Note

Influence of the liquid chromatographic mobile phase on the phase transitions of alkyl-bonded silicas studied by gas chromatography

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Using gas chromatography of a test solute, we previously demonstrated a phase transition for a densely grafted C_{18} silica in the usual temperature range of separations by reversed-phase liquid chromatography[1]. More recently, Kessaissia *et al.* found a similar transition at cryogenic temperatures for alcohols chemisorbed at the surface of silica by the same method[2].

In the previous paper, we suggested that the hydrophobic pressure of the solvent could increase the transition temperature and that such a phenomenon would complicate the often discussed problem of retention by reversed-phase liquid chromatography. This paper supports this hypothesis by means of "inverse" gas chromatographic experiments in which the bonded material is covered with different stationary phases.

In addition, the use of a C_{22} alkyl chain in place of a C_{18} chain leads to more abrupt transitions and sheds some light on the critical influence of the chain length in the chromatographic applications of these materials.

EXPERIMENTAL

The preparation of densely covered alkyl-silicas by means of alkyldimethyl(dimethylamino)silanes according to Kováts' technique[3] has already been described[1]. The intermediate reagent docosyldimethylchlorosilane is distilled at 185°C and $5 \cdot 10^{-3}$ Torr. Docosyldimethyl(dimethylamino)silane is distilled at 185°C and $3 \cdot 10^{-3}$ Torr. The so-called "inverse" gas chromatographic technique using an auxiliary solute and a suitable chromatograph has also been described in earlier papers[1,4].

The stationary phases were commercial products: ethylene glycol (purissimum grade) and *n*-henicosane (purum grade) from Fluka (Buchs, Switzerland), and glycerine (doubly distilled), triethylene glycol (for synthesis grade) and squalane (for gas chromatography grade) from Merck (Darmstadt, G.F.R.).

The silica substrate was Spherosil XOB 015 beads (100–200 μm diameter) from Rhône-Poulenc (Paris, France), with a specific surface area of 27.7 m^2/g (ref. 1). The percentage of bonded carbon was determined by the Service d'Analyse du CNRS (Solaise, France) and corresponded to the coverage of the surface by 4.35 $\mu mol/m^2$ of $C_{22}H_{45}$ radicals.

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RESULTS AND DISCUSSION

C_{22} bonded uncoated silica column

Fig. 1a shows the logarithm of the absolute retention volume of heptane per unit mass of substrate (V_s) as a function of the reciprocal of the temperature ($1/T$) for the C_{22} bonded Spherosil. As for the C_{18}-bonded Spherosil (ref. 1 and Fig. 1b), the line deviates from the linearity usual in both gas–solid and gas–liquid chromatography, except when a stationary phase transformation modifies either the number or the nature of the retention sites.

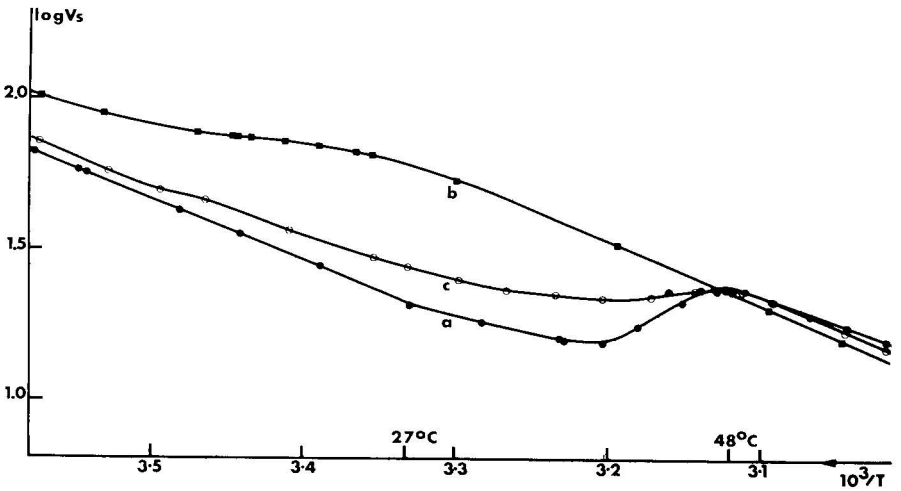

Fig. 1. Variation of log V_s with $1/T$ for *n*-heptane as solute: (a) on C_{22}-bonded (4.32 μmol/m^2) Spherosil XOB 015, (b) on C_{18}-bonded Spherosil XOB 015 (1) and (c) on a less densely (4.18 μmol/m^2) C_{22}-bonded Spherosil XOB 015.

However, for C_{22}-bonded silica the phenomenon is characterized by an overall increase in the retention volume in a limited range of temperature between 40 and 48°C, instead of the limited broad inflection of the curve between 7 and 27°C observed for the C_{18}-bonded material. Thus, better than the results with the C_{18} silica, the new experiments suggest an analogy of this transformation with the condensed phase–expanded phase monolayer transitions of fatty compounds physisorbed at the surface of polar substrates[4–6]. In both instances the increase in retention with increasing temperature is a consequence of a change which gives the solute access to the inner part of the alkyl layer.

These results substantiate the observations of Little *et al.*[7] of the different chromatographic properties of C_{18} and C_{22} alkyl-bonded materials. These differences may result from the physical state of the chains.

However, the marked character of the transition depends critically on the bonding density. If this density is reduced from 4.32 to 4.18 μmol/m^2, the amplitude of the gas chromatographic transition becomes much more limited (Fig. 1c).

Columns of C_{22} bonded silica covered with various liquids

Glycol stationary phases. As Fig. 2a shows for glycerine, the most striking

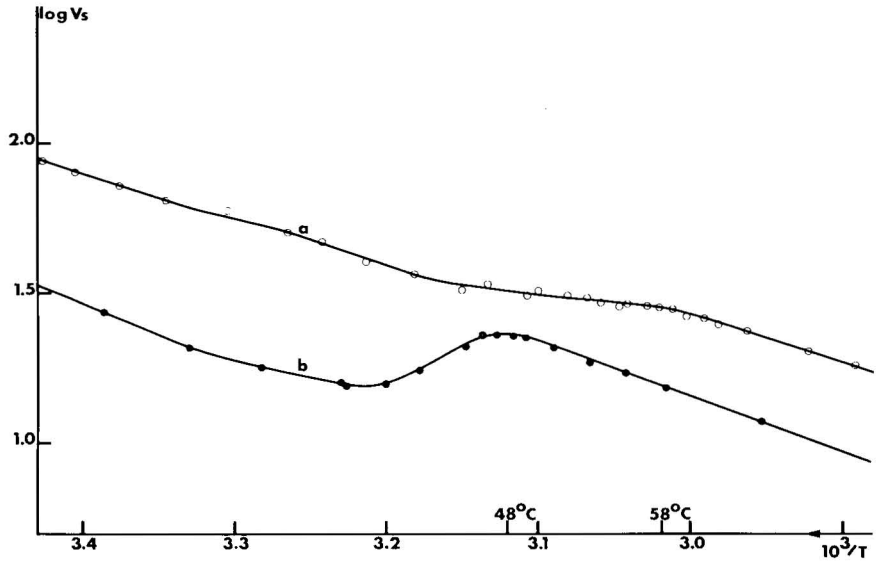

Fig. 2. Variation of log V_s with 1/T for *n*-heptane as solute and (a) 8.9% of glycerol on C_{22}-bonded Spherosil XOB 015 and (b) for uncoated C_{22}-bonded Spherosil XOB 015.

feature with these coated bonded phases is the marked increase in the end-temperature of the transition. In addition, this shift is only large for strongly associated stationary phases (end temperature of the transition: 58°C for glycerine, 57 C for ethylene glycol, 49°C for triethylene glycol). Changes in the ordinate of the curves could easily be ascribed to the retention by the coating stationary phases themselves.

Replacement of glycerine with a glycerine–water mixture does not result in an

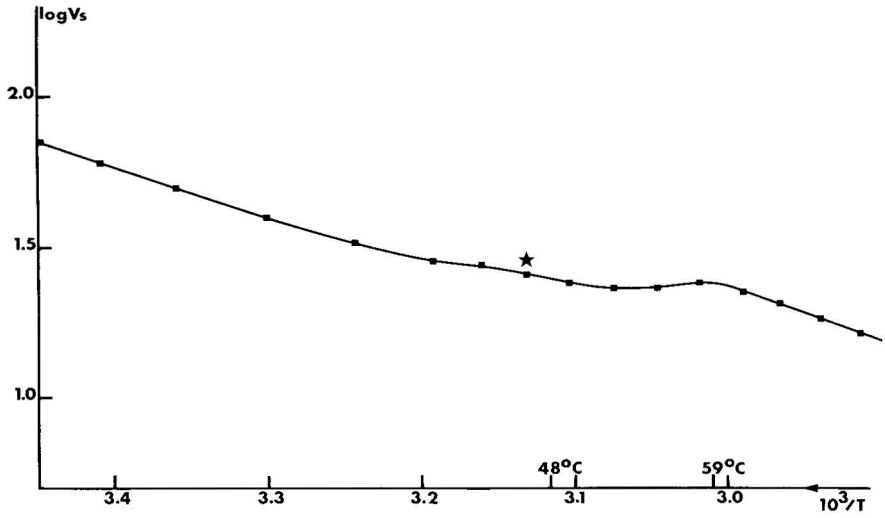

Fig. 3. Variation of log V_s with $1/T$ for *n*-heptane as solute on C_{22}-bonded Spherosil XOB 015 coated with 8.9% of glycerol and 9% of water.

additional shift of the end-temperature of the transition* (Fig. 3). However, a particularly sharp increase in V_s appears about 10 °C above the transition temperature observed for the uncoated bonded silica.

It is worth noting the similarity of the experimental device in two kinds of experiments: first the previous experiments involving mixtures of water and strongly hydrophobic associated compounds and those inside a column in reversed-phase chromatography with water–methanol as the mobile phase. Therefore, it is logical to think that in this sort of chromatography the retention properties of the bonded silica may depend strongly on changes in a very narrow range of temperatures and that this range depends strongly on the composition of the mobile phase.

The only difference lies in the incomplete filling of the pores in the above-described experiments (Fig. 4). Part of the surface is not submitted to the effect of the hydrophilic solvent. This situation explains the appearance of a low secondary transition at the temperature marked by an asterisk in Fig. 3, which is the transition temperature in the absence of a coating stationary phase.

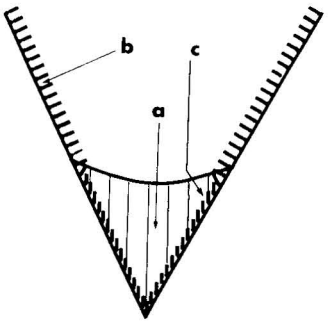

Fig. 4. Model of pore: (a) capillary condensate of liquid stationary phase; (b) bonded phase at the gas–solid interface; (c) bonded phase immersed in the liquid stationary phase.

These results on the influence of strongly hydrophilic liquids substantiate the recent comment of Scott and Simpson[8], after isothermal measurements of reversed-phase retention volumes with water as the mobile phase, that under the influence of water the hydrocarbon chains dispersively interact with themselves and that this interaction results in anomalously low retention volumes.

When polar test solutes are used in place of alkane (Figs. 5 and 6). the transitions are less abrupt, particularly with a coated substrate. However, it is worth noting that an inversion of the solute order may occur across the transition, as shown in Fig. 5 for methyl propyl ketone and dioxane on uncovered bonded silica.

Apolar stationary phases. Although it is unusual to use an non-polar mobile phase with bonded silica in liquid chromatography, replacing hydrophilic liquids with squalane is instructive (Fig. 7). The amplitude of the transition is reduced, but the end temperature of this transition is noticeably lowered (from 48 to 42 °C) at the same time. It is as if there were a solution of squalane in the alkyl layer. In opposition to

* With some reservations owing to the limited efficiency of the water-coated Chromosorb P column used as a saturator, when the temperature rises above 60 °C.

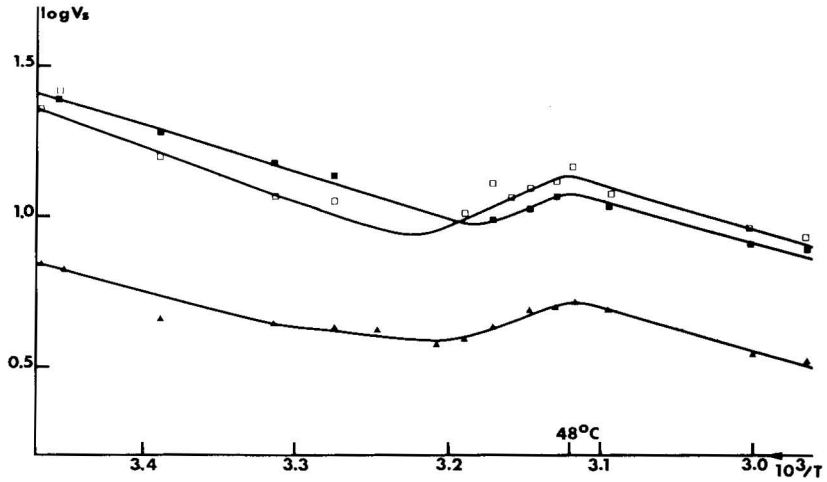

Fig. 5. Variation of log V_s with $1/T$ for uncovered C_{22}-bonded Spherosil XOB 015. Solutes: ■ = dioxane; □ = methyl propyl ketone; ▲ = chloroform.

hydrophilic liquids, squalane reduces the cohesion of the bonded chains and lowers their transition temperature as a result of a cryoscopic-like effect.

Coating the bonded silica with a long-chain *n*-alkane results in a very intriguing material, although it is of no practical use in liquid chromatography at present. At low temperature (in this instance under 60° C), the alkane chains have been inserted into the grafted alkyls to give a very compact mixed monolayer, physisorbed and chemisorbed at the same time. Riedo *et al.*[9] demonstrated such layers by contact angle measurements. We have previously published a study of these phases by inverse gas chromatography for C_{18}-bonded silica and heptadecane[1]. In the most compact

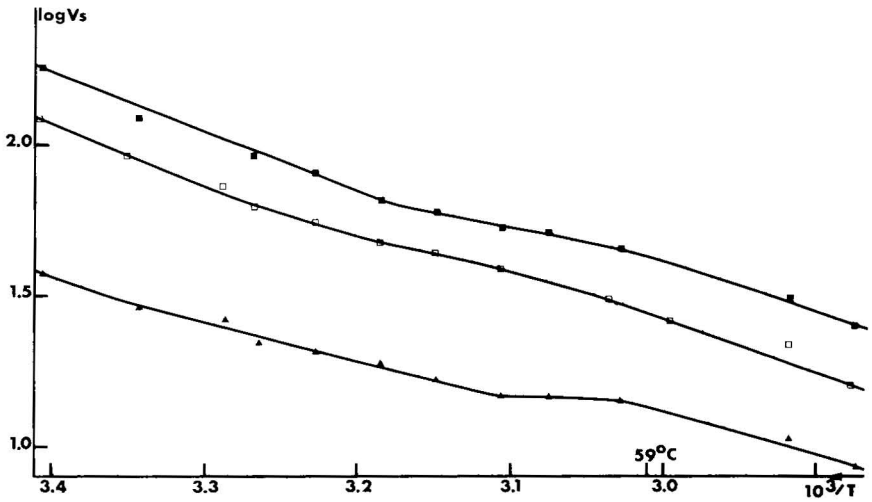

Fig. 6. Variation of log V_s with $1/T$ for 8.9% of glycerol on a C_{22}-bonded Spherosil XOB 015. Solutes: ■ = dioxane; □ = methyl propyl ketone; ▲ = chloroform.

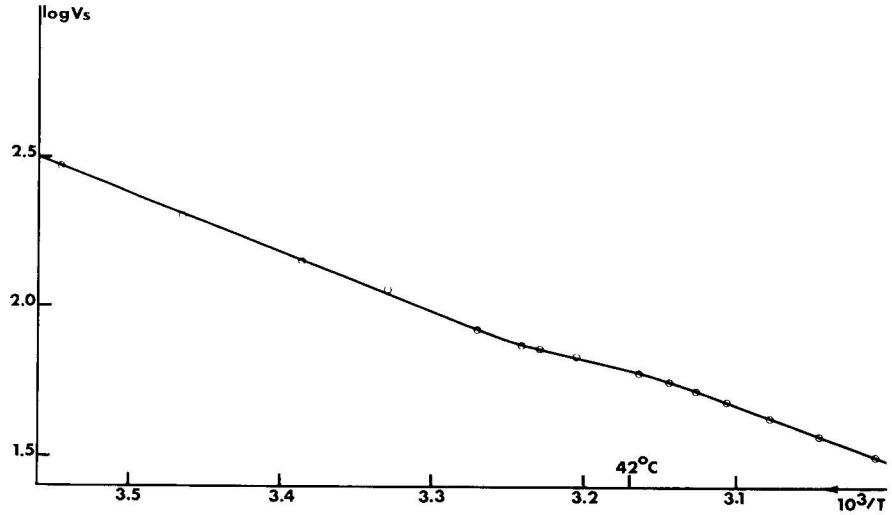

Fig. 7. Variation of log V_s with $1/T$ for *n*-heptane as solute on C_{22}-bonded Spherosil XOB 015 coated with 6.1% of squalane.

layers, the area of substrate occupied for each chain is 0.21 nm^2, as in the classical condensed phases of polar fatty compounds at the surface of water. Such a two-dimensional phase undergoes a melting-like transition at 51°C.

Coating the C_{22} silica with *n*-heneicosane gives rise to very similar phenomena. As Fig. 8 shows, a transition may be observed between about 60 and 68°C, rather sharper at the end. However, it is worth noting that the phenomenon occurs at a much higher temperature than both the transition of the pure bonded silica (51°C) and the melting of heneicosane (40°C; the melting transition of an excess of bulk heneicosane can be seen in Fig. 8 at this temperature). In addition, such layers are not the exclusive property of alkanes and alkyl groups of exactly the same length, as we shall show in a future paper.

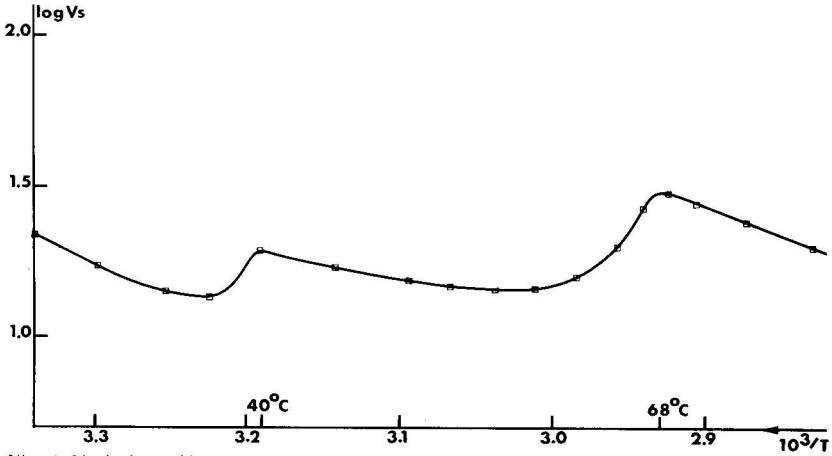

Fig. 8. Variation of log V_s with $1/T$ for *n*-octane as solute on C_{22}-bonded Spherosil XOB 015 coated with 3.1% of *n*-heneicosane.

CONCLUSION

If long-chain alkyl-bonded silicas are immersed in various liquids, the physical state and the chromatographic properties of the bonded material depend very strongly on the temperature. This dependence is all the more critical if the alkyl chain is long ($>C_{18}$) and if the chain coverage is dense (>4 μmol/m^2). However, the temperature range of the transition is raised by a hydrophilic and lowered by a hydrophobic medium (inserted alkane phase excluded).

As the normal transition range of a C_{18}-bonded silica is near room temperature (7–27° C, ref. 1), it is obvious that the retention properties and selectivities of these materials may depend on the temperature and density of bonding in a very complex way. This situation could explain both the irreproducibility of analytical data[10,11] and many controversial points of view in the field of reversed-phase liquid chromatography.

Consequently, we suggest that the choice of C_{18} for economic and practical reasons has not been very good, and slightly shorter or longer chains could have given better results. In addition, it is not at all certain that densely covered silicas would give better analytical results.

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Note

Comparison of C_n bonded silica gel thin-layer chromatographic plates: conditions for use and separations of some barbiturates*

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The advantages of a hydrolytically stable stationary phase, made with small (5–20 μm) silica gel particles chemically bonded with *n*-octyl (C_8) or *n*-octadecyl (C_{18}) groups, are well known in high-performance liquid chromatography (HPLC)[1]. At present, in most papers dealing with separations on columns, these types of bonded coatings are employed in a reversed-phase mode with aqueous–organic modifier eluents. The advantages of aqueous solvent systems include compatibility with biological samples. Also, highly polar compounds, previously separated mainly by means of ion-exchange chromatography, can be separated successfully by reversed-phase (RP) HPLC.

Similar advantages exist for reversed-phase thin-layer chromatography (RP-TLC) carried out on chemically modified silica gel-coated plates. However, there have been few TLC studies using this type of bonded coating material[2–5], although the performance of TLC with reversed-phase plates has been studied[6] and examples of analyses have been published[7]. The use of RP-TLC to assess hydrophobicity in studies of quantitative structure–activity relationships for drugs[8] and to correlate R_M values with the structures of barbiturates[9] have also been reported.

Another potentially useful application of bonded plates arises from their analogous behaviour to RP-HPLC on columns[10–12]. In fact, using RP-TLC, many eluting systems can be tested inexpensively and in a relatively short time, and these preliminary results may be easily adapted to RP-HPLC on columns.

As an extension of previous work[13] and in order to widen the application of RP-TLC in the analysis of drugs and/or biological materials, in this work we investigated the behaviour of some commercially available reversed-phase silica gel precoated plates, in connection with some properties of the eluent, *viz.*, the type of organic modifier, the percentage of water, the pH and the ionic strength. With a view to possible applications, the experimental conditions were chosen so as to shorten the development time as much as possible.

In addition, the separation of mixtures of barbituric acid derivatives is reported. Numerous papers on the separation of barbiturates by conventional TLC have been published[14–17]; nevertheless, the very short time of separation (about 15 min)

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and the high reproducibility of the R_F values without any special experimental precaution seemed to be noteworthy.

EXPERIMENTAL

Compounds

Table I lists the barbiturates examined. Because of the hydrophobicity of RP-8 and RP-18 layers, methanol was selected as the solvent; 0.1–1 % methanolic solutions of samples were employed.

Thin-layer chromatography

Plates. Different types of chemically bonded silica gel pre-coated plates, all 5 × 5 cm and containing a fluorescent indicator, were tested: HPTLC F_{254} RP-8 and RP-18 (Merck, Darmstadt, G.F.R.); Stratocrom Si F_{254}-C_{18} W (made by Whatman for Farmitalia Carlo Erba, Milan, Italy); and RP OPTI-UP C_{12} (Antec, Bennwil, Switzerland) (for preliminary experiments only). No pre-treatment of the plates before use was performed.

Eluents. Mixtures containing from 10 to 90 % (v/v) of an organic modifier and water (or aqueous solutions) were employed. The organic modifiers were methanol, acetonitrile, isopropanol and tetrahydrofuran. Aqueous solutions were 0.05–1 *M* lithium chloride, 0.2 *N* hydrochloric acid, 0.1 *N* acetic acid, 0.1 *N* ammonia solution and 0.1 *N* sodium hydroxide. Buffer solutions were 0.05 *M* borax + 0.1 *N* sodium hydroxide (pH 10.32) and 0.1 *M* potassium dihydrogen orthophosphate + 0.1 *N* sodium hydroxide (pH 6.11).

Spotting and development. To achieve high resolutions, the spot area must be very small[18], and in this work it was usually about 2 mm. Portions of 0.2–0.3 μl of 0.1–1 % methanolic solutions of samples were applied 1 cm from the lower edge of the plate using a Hamilton syringe (1 μl) in connection with a micrometer. Rapid saturation of the developing chamber can be achieved by employing a glass jar, as small as is compatible with the size of the plate (10.5 cm high × 6.5 × 6.5 cm) and equipped with a ground-glass stopper. Standardized conditions for dipping the plates in the eluent were found to be very useful: a height of about 0.7 cm of eluent gave a short elution time without affecting the resolving power. The eluent was placed in the glass jar *ca.* 30 min prior to insertion of the plates. Ascending development was carried out at room temperature for 3.8 cm.

Detection. After development, the wet plates were exposed to an ammonia atmosphere for 5 min and the spots of the barbiturates were located under ultraviolet light by quenching of fluorescence at 254 nm[19]. A KM-3 chromatogram spectrophotometer (Carl Zeiss, Oberkochen/Wurttemberg, G.F.R.), in the reflectance mode, was used to measure directly barbiturate spots after separation on RP-18 plates. The plates were scanned in a direction parallel to that of the solvent flow.

RESULTS AND DISCUSSION

Particular attention was paid to the chromatographic conditions in order to obtain a development time of less than 40 min in all the experiments and, at the same time, a high resolution. As mentioned under Experimental, development to a height

of about 4 cm was sufficient to obtain a good resolution of a mixture of four or five compounds having R_F values in a limited range (*e.g.*, all between 0.25 and 0.65).

Eluents and development time

In all the experiments described below, the barbiturates reported in Table I were studied. Figs. 1–3 show the dependence of the development time on the composition of the mobile phase (type of organic modifier and percentage of water) for some commercially available reversed-phase pre-coated plates. Four organic modifiers with increasing polarity and chosen from those most commonly used in HPLC were investigated as eluents.

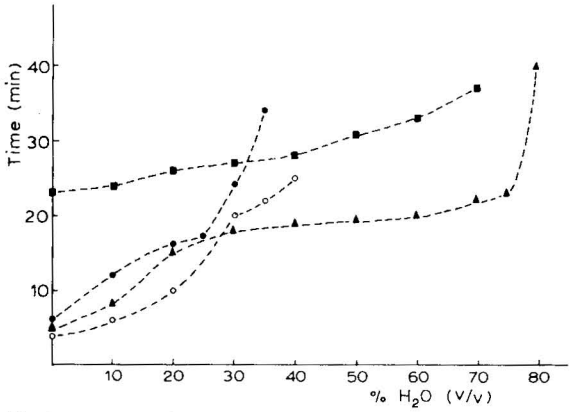

Fig. 1. Influence of the percentage of water in the eluent on the development time using HPTLC F_{254} RP-18 (Merck) silica gel pre-coated plates. Plates: 5 × 5 cm. Ascending development for 3.8 cm. ■, Isopropanol; ▲, tetrahydrofuran; ●, ethanol; ○, acetonitrile.

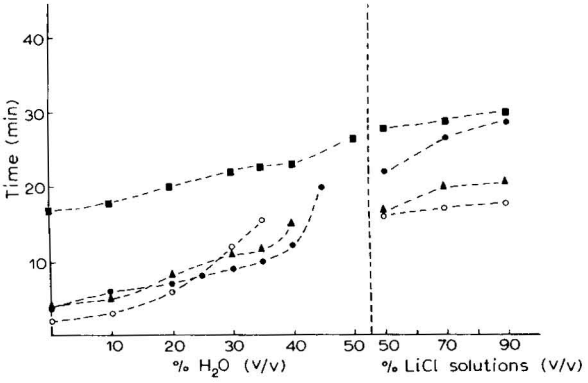

Fig. 2. Influence of the percentage of water in the eluent on the development time using Stratocrom Si F_{254}–C_{18} W (Carlo Erba) silica gel pre-coated plates. Chromatographic conditions and symbols as in Fig. 1.

It can be seen that the results depended on the type of plate used, and probably the binder material plays an important role. With Merck RP-18 plates (Fig. 1), when using methanol–water or acetonitrile–water mixtures as eluents, the elution time increased rapidly with increasing water content; 35–40% of water seems to be the upper

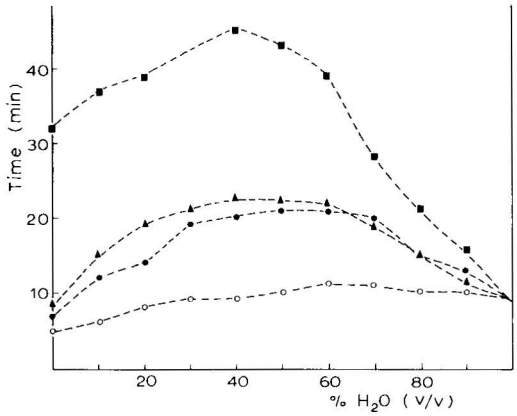

Fig. 3. Influence of the percentage of water in the eluent on the development time using RP OPTI-UP C_{12} (Antec) silica gel pre-coated plates. Chromatographic conditions and symbols as in Fig. 1.

limit if an elution time longer than 40 min is to be avoided. Although under our experimental conditions a higher percentage of water than previously reported[2,7] may be employed, the range of possibilities was always limited. Shorter elution times were obtained with Carlo Erba plates by employing the same eluent mixtures (Fig. 2), but the limits in use seemed to be about the same: the silica gel layer became detached from the glass support when eluents with water content higher than 50% were used. Nevertheless, it is interesting that this damage to the layers could be avoided by replacing the water in the eluent with lithium chloride solutions of increasing concentration: 0.1 *M* with 50% of methanol or acetonitrile and up to 1 *M* with 10% of methanol or acetonitrile. The elution time always remained below 30 min (Fig. 2).

The same experiments were carried out on Merck RP-8 plates. For both methanol–water and acetonitrile–water eluents our results were in agreement with those of Siouffi *et al.*[7]; the development time was shorter for RP-8 than RP-18 plates.

As eluents containing more than 50% of water are often employed in HPLC on columns, other organic modifiers were tested. Good results were achieved by employing Merck RP-8 or RP-18 plates and isopropanol– or tetrahydrofuran–water as eluent. Up to 70–80% of water can be used (Fig. 1). This result was interesting because of the different properties of tetrahydrofuran compared with the other solvents tested[20]. With the Carlo Erba plates, the addition of lithium chloride is always necessary if isopropanol or tetrahydrofuran containing more than 50% of water must be employed (Fig. 2). However, tetrahydrofuran did not seem to be a good eluent for this type of plate, because of the elongated and irregular shapes of the spots.

Only preliminary experiments were carried out on OPTI C-12 plates. So far as the amount of water in the eluent is concerned, OPTI C_{12} plates behave differently (Fig. 3) from those described above. For all the four organic modifiers tested, any percentage of water could be used. The elution time was always very short, with a maximum and a subsequent decrease with increasing water percentage. This difference in behaviour could be ascribed to the capacity of this type of layer to absorb water. The sequence of elution time, however, agrees with that found for C_{18} and C_8 plates; isopropanol–water mixtures showed the longest elution times. Although com-

plete results cannot be given for OPTI C_{12} plates, their behaviour seemed to be interesting. The very short elution time and the possibility of utilizing any ratio between the organic modifier and the water in the eluent (Fig. 3) are very advantageous properties.

pH of eluent. No damage to any of the four types of plates was observed on employing strongly acidic or basic eluent mixtures [*e.g.*, methanol–0.2 *N* hydrochloric acid (6:4) or methanol–1 *N* sodium hydroxide (6:4)]. Probably this favourable result depended on the short development time. It is important to note that with respect to HPLC on columns, the range of pH of the eluents widened, mainly on the basic side.

R_F values

The Merck and Carlo Erba plates gave good reproducibility of the R_F values (relative standard deviation of R_F values = 0.02). In addition, the R_F values of the eight barbiturates under examination obtained on the two different types of RP-18 plates compared very well. No significant difference in the R_F values were found on employing as eluents mixtures containing a constant ratio of methanol or acetonitrile and lithium chloride solution of increasing concentration (0.1–1 *M*).

In general, two practical advantages were found by working with reversed-phase plates rather than conventional plates: first, no particular attention need be paid to the operating conditions with respect to the effect of ambient moisture on the layer, and second, there is the possibility in many instances of reusing the plates after washing them. Ascending chromatography with methanol as eluent gave satisfactory results and no change in performance was noted.

Separation of barbiturates

Table I gives R_F values for some barbiturates obtained with Merck and Carlo Erba RP-18 plates. To investigate the effect of the eluent on the R_F values, various mixtures of each of the four organic modifiers and water, in different ratios, were tested. In all instances the R_F values of each of the eight barbiturates decreased as the amount of water in the eluent increased and the sequence of the R_F values was the same for whatever organic modifier. Mixtures of organic modifier and aqueous acetic acid or ammonia, phosphate or borax buffer were also tested. A difference in R_F values occurred on working at basic or acidic pH (Table I). This difference probably depends on the predominance of the keto or enol form of the barbituric acid derivatives, the pK_a values being reported for some of these compounds in the range 7—8 (ref. 21).

The best separations were achieved by employing methanol and phosphate or borax buffer in the ratio 3.5:2 or isopropanol and the same buffer (1:2) (Figs. 4 and 5). A shorter elution time (about 15 min) was obtained by employing Carlo Erba plates and a better resolution by using the Merck plates.

For the possible use of these results in the screening of biological samples, it is interesting to note that the long-acting phenobarbital (compound 2) was separated from the short- to intermediate-acting amobarbital and secobarbital (compounds 7 and 8, respectively). In addition, as reported for conventional TLC[22], the speed of action of barbiturates seemed to be related to their R_F values: in our work, the faster acting barbiturates had lower R_F values than the slower acting compounds.

By means of the experiments described, 0.7 μg of phenobarbital and 0.2–0.3 μg of the other barbiturates can be detected.

TABLE I

R_F VALUES FOR BARBITURATES

Eluents: 1 = methanol–0.1 *M* acetic acid (3.5:2); 2 = methanol–phosphate buffer (3.5:2); 3 = methanol–0.1 *M* ammonia solution (3.5:2); 4 = isopropanol–borax buffer (1:2).

No.	*Barbituric acid*	*Trivial name*	*Proprietary name*	*R_F value*			
				Eluent 1	*Eluent 2*	*Eluent 3*	*Eluent 4*
1	5,5-Diethyl-	Barbital	Veronal	0.53	0.59	0.72	0.58
2	5-Phenyl-5-ethyl-	Phenobarbital	Luminal	0.41	0.50	0.63	0.40
3	5,5-Diallyl-	Diallylbarbital	Dial	0.43	0.51	0.60	0.41
4	5-(1-Cyclohexenyl)--5-ethyl-	Cyclobarbital	Phanodorm	0.32	0.38	0.52	0.31
5	5-Allyl-5-isobutyl-	Itobarbital	Sandoptal	0.33	0.36	0.48	0.30
6	5-(1-Cycloheptenyl)--5-ethyl-	Heptabarbital	Medomin	0.25	0.31	0.42	0.26
7	5-Ethyl-5-(3-methylbutyl)-	Amobarbital	Amital	0.23	0.28	0.39	0.20
8	5-Allyl-5-(1-methylbutyl)-	Secobarbital	Seconal	0.22	0.26	0.35	0.18

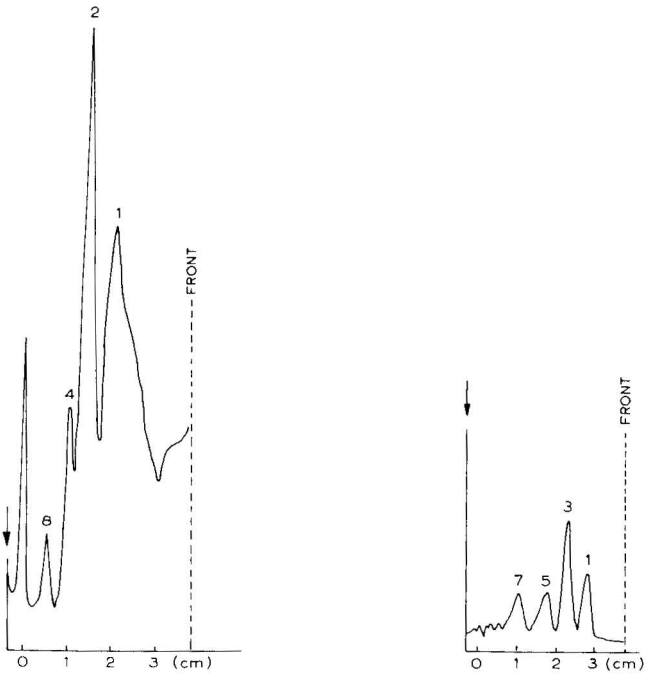

Fig. 4. Scan of some barbiturates separated on chemically bonded silica gel pre-coated plates (RP-18). Eluent: isopropanol–phosphate buffer (1:2, v/v). Ascending development to a height of 3.8 cm. Wavelength, 254 nm; slit width, 3.5 mm; v_p = 50 mm min^{-1}; v_c = 60 mm min^{-1}. Compounds: 1 = barbital; 2 = phenobarbital; 4 = cyclobarbital; 8 = secobarbital. v_p = plate travel; v_c = recorder chart speed.

Fig. 5. Scan of some barbiturates separated on chemically bonded silica gel pre-coated plates (RP-18). Eluent: methanol–borax buffer (3.5:2, v/v). Ascending development to a height of 3.8 cm. Wavelength, 254 nm; slit width, 0.5 mm; slit length, 3.5 mm; v_p = 100 mm min^{-1}; v_c = 100 mm min^{-1}. Compounds: 1 = barbital; 3 = diallylbarbital; 5 = itobarbital; 7 = amobarbital.

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Note

Direct gas chromatographic determination of the products of catalytic air oxidation of *n*-butene-1 to maleic anhydride in the gaseous reaction mixture

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Catalytic gas phase oxidation of *n*-butene-1 with air yields a large number of oxidation products in addition to maleic anhydride (MA), butene isomers and butadiene resulting from oxidative dehydrogenation. These are mainly furan, crotonaldehyde, acetic acid, propionic acid, acrylic acid, α-methylacrylaldehyde, ethyl methyl ketone, butanedione, isobutyraldehyde, water, carbon monoxide and carbon dioxide. Up to now, the analysis of these reaction products has been mostly carried out in such a way that higher boiling products are condensed and/or are subjected to absorption from the reaction gas by suitable solvents before gas chromatographic analysis of the remaining gaseous components. This procedure is mainly used because of the high boiling and melting point of MA, as severe problems arise when the gaseous reaction mixture is introduced into the gas chromatograph.

However, this procedure is disadvantageous when separating MA and water by condensation from the gas because at least part of MA will be hydrolysed to maleic acid. The problem is even more severe when water is used as an absorbent. However, under the usual conditions the resulting maleic acid is not amenable to subsequent direct gas chromatographic analysis. Hence, the determination of MA is performed following complete hydration to maleic acid, mainly by potentiometric titration, whereas the other products are determined by gas chromatography. It is known, however, that MA can be determined directly by gas chromatography on various columns with stationary phases consisting of phthalic acid ester[1,2], silicone oil[2], cyanosilicone oil[3] or Porapak Q[4,5]. Columns with polyesters[6] and di(2-ethylhexyl) sebacate + sebacic acid[7] as stationary phases are also applicable to the gas chromatographic determination of MA.

A method is described below that allows the direct quantitative gas chromatographic analysis of the products of the gas-phase oxidation of *n*-butene-1 ("direct method"), without the need to analyse any condensate or absorbent for these products ("indirect method").

GAS CHROMATOGRAPHIC ANALYSIS

In the oxidation of *n*-butene-1 the resulting gaseous products are divided into two fractions. One fraction is passed through two cold traps filled with ice, in which

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TABLE I

CONDITIONS FOR GAS CHROMATOGRAPHIC ANALYSIS OF GASEOUS PRODUCTS FROM OXIDATION OF *n*-BUTENE-1

Separation column	*Conditions for analysis*	*Detector*	*Substances*	*Retention time (sec)*
Molecular sieve 13X (100–200 mesh), 1 m × 3 mm I.D.	He, 25 ml/min, room temperature, isothermal, 2-ml sample loop	Thermal conductivity	CO	190
Dimethylsulpholane on Chromosorb (30–60 mesh), 5 m × 3 mm I.D.	He, 25 ml/min, room temperature, isothermal, 2-ml sample loop	Thermal conductivity	CO_2	150
			n-Butene-1	300
			trans-Butene-2	360
			cis-Butene-2	402
			Butadiene	540
Di(2-ethylhexyl) sebacate + sebacic acid on Chromosorb W AW, 2 m × 2 mm I.D.	N_2, 25 ml/min, 120°C, isothermal, detector 200°C, injector 200°C, inlet 150°C, 1-ml sample loop	Flame ionization	C_4 hydrocarbons	44
			Furan	69
			Isobutyraldehyde	90
			α-Methylacrylaldehyde	97
			Butanone	109
			Crotonaldehyde	172
			Acetic acid	251
			Propionic acid	480
			Acrylic acid	618
			MA	1050

the higher boiling products are condensed. The remaining gaseous products (butenes, butadiene, carbon monoxide and carbon dioxide) are analysed on a dimethylsulpholane–chromosorb column and on a molecular sieve column. The second fraction is cooled only to 150°C and passed via a heated tube directly to a heated gas sample loop of the gas chromatograph; the remaining products are analysed on a di(2-ethylhexyl) sebacate + sebacic acid–Chromosorb column. Equilibrium calculations for the hydration of MA have shown that even in the presence of water vapour under the above conditions virtually no maleic acid is formed. Table I gives the analytical conditions together with retention times of the different components. Fig. 1 illustrates a typical chromatogram for the oxygenated derivatives of *n*-butene-1 together with the total C_4 hydrocarbons.

For the quantitative evaluation of the chromatogram, the necessary response factors for the liquid products under ambient conditions or at higher temperatures were determined in such a way that a carrier gas stream was loaded with pure substance and introduced into the gas chromatograph for calibration. In each instance the

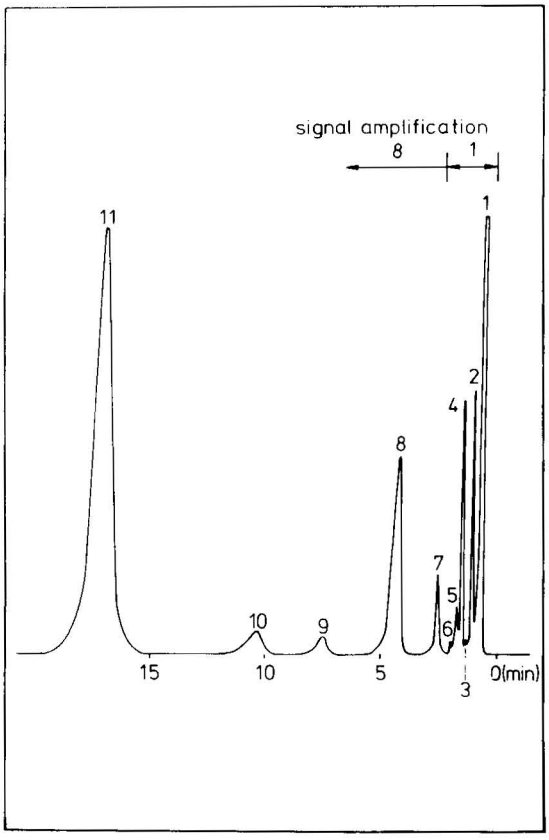

Fig. 1. Chromatogram of a characteristic gaseous reaction mixture produced by air oxidation of *n*-butene-1 to MA. Concentration of *n*-butene-1 at reactor inlet: 1.5 mole-%. Identities of peaks, with yields in wt.-% in parentheses: 1 = C_4 hydrocarbons (65.8); 2 = furan (3.1); 3 = isobutyraldehyde (0.1); 4 = α-methylacrylaldehyde (4.1); 5 = ethyl methyl ketone (0.9); 6 = butanedione (0.3); 7 = crotonaldehyde (0.5); 8 = acetic acid (2.5); 9 = propionic acid (0.1); 10 = acrylic acid (0.3); 11 = MA (13.1).

prevailing concentrations in the gas stream were determined with the help of the vapour pressure according to ref. 8. Saturation was achieved by first bubbling the carrier gas through the liquid and subsequently passing it through a reflux condenser having a temperature of 10–15°C below that of the liquid so that part of the evaporated liquid is re-condensed. The saturation pressure corresponds to the condenser temperature, which could be accurately measured. The response factors, f, obtained in this way on the basis of propionic acid are compared in Table II for four important *n*-butene-1 oxidation products (MA, acrylic acid, propionic acid and acetic acid), with values being determined by the gas chromatographic analysis of acetone solution or by calculation according to ref. 9 by $f = M_i/(n_C \cdot 12)$, where M_i is the molecular mass of i and n_C is the number of "available" carbon atoms of i).

For the remaining components of a typical product gas no response factors were experimentally determined, as they were present either in only minute concentrations below the limit of determination (ethyl methyl ketone, butanedione and isobutyraldehyde) or could not be analysed quantitatively owing to polymer formation (α-methylacrylaldehyde, crotonaldehyde and furan) under the conditions of gas chromatography used. For these reaction products the response factors are actually estimated according to ref. 9 in order to obtain at least some indication of the magnitude of their concentrations.

The response factors measured for MA and propionic acid, as is evident from Table II, agree well with the calculated values[9]. On the other hand, large deviations for acetic acid and especially for acrylic acid may possibly be caused by polymerization, which can occur at elevated temperatures. Direct determination of the MA content in a product gas by gas chromatography corresponds, within the limits of experimental accuracy, with the value obtained when MA was first absorbed in acetone and water and then the MA content was determined by potentiometric titration of maleic acid in water and gas chromatographic determination of MA dissolved in acetone. It should be mentioned, however, that the response factor for MA in acetone solution derives from the gas phase.

The linearity of the response factors with respect to concentration was examined over the range 0.05–0.8 mole-% for each substance; the result for MA is shown in Fig. 2. After about 100 analyses of the product gas, the estimated response

TABLE II

RESPONSE FACTORS OF MONOCARBOXYLIC ACIDS AND MA BASED ON PROPIONIC ACID

For separating column and conditions for analysis, see Table I.

Reaction product	*Response factor*		
	Direct (gas phase)	*Indirect (acetone solution)*	*Calculated*[9]
Acetic acid	1.50 ± 0.03	1.75 ± 0.05	1.62
Propionic acid	1.0	1.0	1.0
Acrylic acid	1.26 ± 0.03	1.40 ± 0.05	0.97
MA	1.26 ± 0.02	1.30 ± 0.10	1.32

factors were re-checked to establish whether the separating properties of the gas chromatographic columns had changed. The measured deviations were within the standard deviation of the response factors.

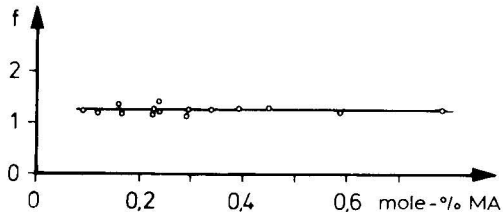

Fig. 2. Response factor of MA based on propionic acid as a function of gas loading with MA.

CONCLUSION

The direct gas chromatographic determination of the products in the gaseous mixture obtained by air oxidation of *n*-butene-1 needs less time than the "indirect method" involving titration of maleic acid and gas chromatographic determination of the other products. The accuracies of the two methods are comparable. The direct method is suitable for establishing quickly and simply the resulting intermediates in the gas phase, *i.e.*, butadiene, furan and crotonaldehyde, which occur at higher concentrations on incomplete conversion of *n*-butene-1, and impurities in MA, especially monocarboxylic acids, which occur on complete conversion of *n*-butene-1.

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Note

Separation of anthocyanin chalcones by high-performance liquid chromatography

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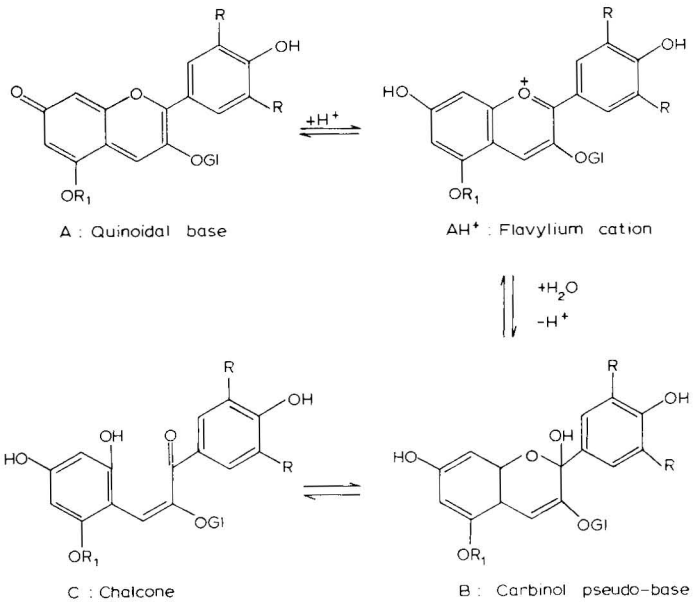
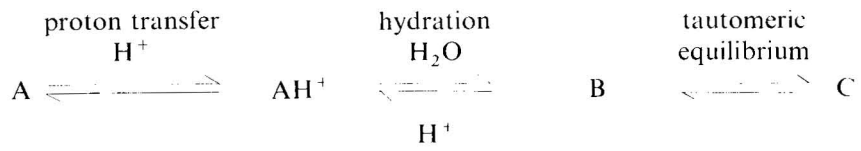
Anthocyanins such as malvidin 3-glucoside and malvidin 3,5-diglucoside can occur in four chemical structures between pH values 0–6, *viz.* the flavylium cation (AH^+), the quinoidal base (A), the carbinol base (B) and the chalcone (C) (Fig. 1)[1,2].



Malvidin 3-glucoside: Gl = glucose, R = OCH_3, R_1 = H
Malvidin 3,5-diglucoside: Gl = R_1 = glucose, R = OCH_3

Fig. 1. Anthocyanin structures.

These structural forms are inter-related according to the following scheme:



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Their proportions at equilibrium are determined by the environmental conditions (pH, temperature, etc.)[3]. Kinetically, the proton transfer and hydration equilibria can be regarded in relative terms as very fast and fast respectively, since relaxation times of malvidin 3-glucoside equilibria at 25°C are of the order of 10^{-5} sec for proton transfer and 1 sec for the hydration equilibria[4]. In contrast, equilibration between the carbinol base (B) and the chalcone (C) of malvidin 3-glucoside is slow, about 1 h at 25°C[4]. To date, the chalcone forms of malvidin 3-glucoside and malvidin 3,5-diglucosides have not been studied in isolation because of the co-occurrence of one or more of the other structural forms, usually in large excess. A method of evaluating the UV–visible absorption spectrum of the chalcone of malvidin 3-glucoside has been described based on measurements of absorbance changes occurring after rapid pH adjustment of an aqueous solution of this anthocyanin from pH 5 to pH < 1 (ref. 4). The calculated spectrum exhibited a broad peak at 350 nm, but the amount of chalcone present was small (*ca.* 10%) compared with the amount of other structures present, *viz.* the carbinol base at pH 5 and the flavylium cation at pH < 1. We have now demonstrated that the chalcone forms of anthocyanins can be separated during the time-scale of high-performance liquid chromatography (HPLC) because of the relatively slow rates of their equilibration with the carbinol forms. For the first time the chalcones of malvidin 3-glucoside and malvidin 3,5-diglucoside have been obtained practically free of the other structures and in sufficient amounts to measure their UV–visible absorption spectra directly and to follow their conversions to their corresponding flavylium cations.

EXPERIMENTAL

Malvidin 3,5-diglucoside was a commercial sample (Fluka, Buchs, Switzerland) and malvidin 3-glucoside was isolated in this laboratory from the skins of red grapes (*Vitis vinifera*). A Spectra-Physics SP8000B high-performance liquid chromatograph was used with its detector operating at 280 nm. A Pye-Unicam LC-UV variable-wavelength detector was used for simultaneous monitoring at other wavelengths. The reversed-phase column (100 × 5 mm) was slurry-packed with Spherisorb Hexyl, 5 μm. The 20-μl samples were filtered through a 0.45-μm Millipore filter before injection. Separations were carried out at a column temperature of 35°C.

Solvent A was 0.60% perchloric acid (pH 1.5), prepared by addition of 60% $HClO_4$ to deionized and glass-distilled water. Solvent B was methanol, glass-distilled from KOH. Both solvents were filtered through a 0.45-μm Millipore filter before use.

Solutions of malvidin 3-glucoside and malvidin 3,5-diglucoside (2.0 and 1.5 mg/ml, respectively, in 0.02 *M* potassium hydrogen tartrate, pH 3.5) were left to stand overnight at room temperature in the dark to attain equilibrium before chromatography. The gradients used for separation of the chalcone glycosides from the equilibrium mixtures were as follows: for malvidin 3-glucoside, 20% of B to 30% B in 15 min; for malvidin 3,5-diglucoside, 2% B to 20% B in 10 min, then 20% B to 60% B in 40 min. Mean retention times for peaks detected at 280 nm were: malvidin 3-glucoside, chalcone glycoside form, 513 sec, flavylium form, 630 sec; malvidin 3,5-diglucoside, chalcone glycoside form 852 sec, flavylium form 1073 sec.

To identify the chalcone glycoside peaks successive separations were carried out using detectors monitoring simultaneously at 280 nm and 525 nm, then 280 nm

and 340 nm. The chalcone glycosides showed no absorbance at 525 nm, but a substantial absorbance at 340 nm. Confirmation was obtained by injecting samples immediately after heating to 100°; the chalcone glycoside peaks were now considerably enlarged, while peaks from the flavylium forms of the anthocyanins were slightly reduced.

Once identified, the chalcone glycoside peaks were collected immediately after passage through the 280-nm detector, in a silica spectrophotometer cell (10 mm pathlength, 0.5 ml capacity) and were transferred at once to the cell compartment (at 28°C) of a Pye Unicam SP8-100 spectrophotometer; their UV–visible spectra between 700 and 210 nm were recorded without delay, using a reference of 75% solvent A, 25% solvent B for malvidin 3-glucoside, and 79% solvent A, 21% solvent B for malvidin 3,5-diglucoside. The relaxation times of the chalcone glycosides were determined by scanning the UV–visible range continuously until negligible change between successive scans was observed. Scanning speed was 2 nm/sec, and the interval between the start of successive scans was 347 sec. For samples requiring minimum exposure to light, continuous scanning was not employed, and absorbance values at 340 nm and 524 nm were read directly from the spectrophotometer at 5-min intervals. Between readings the samples remained in the instrument with the incident radiation masked off.

RESULTS AND DISCUSSION

Usually, anthocyanins are separated by HPLC as their coloured flavylium cations and are monitored at their peak wavelengths (*ca.* 525 nm). Peak shape is affected by solvent pH. At pH 2.5, appreciable quantities of the carbinol forms are also present, which broaden the visible peaks because of interconversion of the two pigment forms on the column[5]. Reducing solvent pH to about 1.5, this being the lower limit of pH stability of the reversed-phase packing materials in use, reduces the amounts of interfering carbinol bases present and sharpens the peaks of the cationic forms. Under such conditions anthocyanins have been separated and identified in plant materials and fruit products. But it should be emphasized that because of rapid interconversion of the various pigment forms induced by pH changes, the quantities of flavylium cations found are determined by the pH conditions pertaining during the chromatographic separation and measurement (about pH 1.5) and may be very different from those in the original plant extract or solution if this is of higher pH.

Anthocyanins also absorb in the ultraviolet region (*ca.* 280 nm). During routine HPLC examination of a solution of malvidin 3-glucoside (pH 3.5), monitoring at 280 nm as well as 525 nm, a small peak was observed at 280 nm which preceded that due to the cationic form. There was no corresponding peak at 525 nm, but a peak appeared when the wavelength of monitoring was changed from 525 nm to 350 nm. It seemed possible that the new peak was due to the chalcone form of malvidin 3-glucoside. To test this, a solution of malvidin 3-glucoside (pH 3.5) was rapidly heated to 100°C. It is known that all the reactions in the scheme from left to right are endothermic, so that heating should shift all equilibria towards the chalcone[2]. Chromatography of the hot solution resulted in the size of the new peak (at 350 nm) being more than doubled while the peak (280 nm) due to the cationic form was reduced. The heated solution was allowed to cool and was re-examined after five days. The new

peak was reduced and the cationic peak was increased to the sizes observed originally; both peaks were identical to those found in the original unheated five-day old solution. Thus the findings were consistent with those expected of the chalcone[2,4]. Similar results were found when a solution of malvidin 3-glucoside at pH 4.5 was treated in the same way.

The new peak was sufficiently well separated from the cationic peak to be collected in a micro-cell and its spectrum scanned as rapidly as possible in a separate spectrophotometer. The spectrum exhibited a broad peak near 340 nm, and a minimum at 274 nm in a solvent containing 75% of 0.60% perchloric acid and 25% methanol; this spectrum is very similar to that computed for the chalcone of malvidin 3-glucoside in aqueous hydrochloric acid by Brouillard *et al.*[4]. When the spectrum was scanned continuously, the broad peak near 340 nm slowly reduced in size with the emergence of more intense peaks at 524 nm and 276 nm, indicating that the chalcone was converting to the cationic form of malvidin 3-glucoside.

The chalcone of malvidin 3,5-diglucoside was also separated and collected after HPLC of a sample of hot solution (pH 3.5). Its spectrum was similar to that of malvidin 3-glucoside chalcone with a trough at 274 nm and a broad peak near 335 nm. The chalcone converted to the flavylium cation of malvidin 3,5-diglucoside ($\lambda_{max.}$ 522 nm and 274 nm) in similar fashion to that observed with malvidin 3-glucoside (Fig. 2). Re-injection of the converted solution into the chromatograph confirmed that the flavylium cation present was entirely malvidin 3,5-diglucoside and that no hydrolysis to either of the malvidin monoglucosides had occurred. The very small absorbance in the vicinity of 522 nm in the spectrum of freshly separated chalcone (Fig. 2) can be attributed to some slight conversion occurring during the time interval between its separation on the column and its initial spectral measurement.

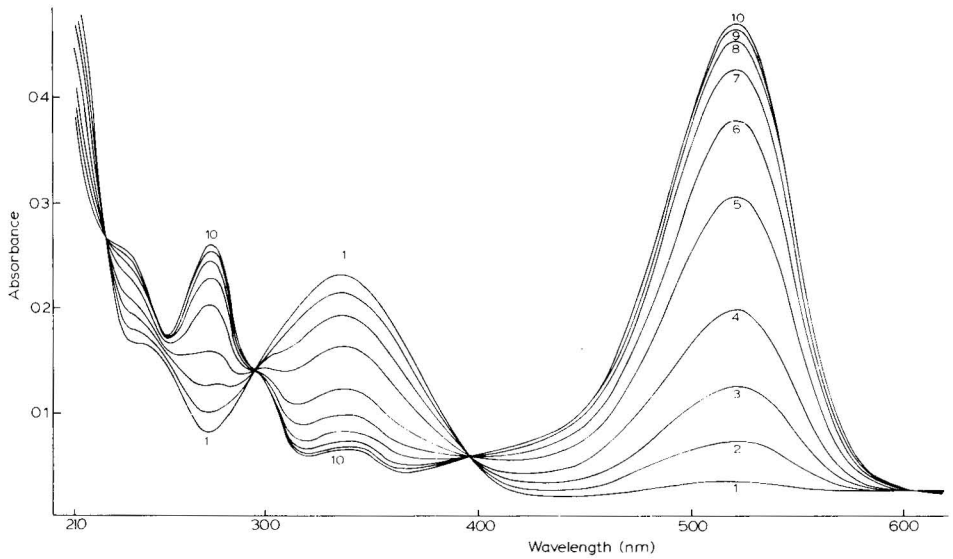

Fig. 2. Absorbance spectra of the chalcone of malvidin 3,5-diglucoside during its conversion to the flavylium cation (solvent 79% A, 21% B). Scan 1: chalcone measured immediately. Scans 2–10: chalcone conversion to the cation. Scans were at intervals of 5.8 min except the interval between scans 4 and 5 which was 7.4 min.

When the isolated chalcone of malvidin 3-glucoside was kept dark and exposed to light only during absorbance measurements made at 340 nm and 524 nm, the rate of chalcone loss (absorbance at 340 nm) and the rate of formation of flavylium cation (absorbance at 524 nm) were as shown in Fig. 3. However, the curves constructed from similar data obtained from the spectral scans (from 700 nm to 210 nm) exhibited initial delays between scans 1 and 3. This phenomenon (Fig. 4) was observed with both anthocyanins. A possible explanation is as follows. From consideration of the work of Jurd[6] and McClelland and Gedge[7] on flavylium salts, it is to be expected that the chalcone isomer from which the anthocyanin cation is derived (via the carbinol base) is the *cis*, this isomer alone having the correct configuration for cyclisation. In contrast the isomer likely to occur at equilibrium is the *trans*. Conversion of *trans* to *cis* should be endothermic. It is conceivable that heating an equilibrated solution of an anthocyanin at pH 3.5 produces a mixture containing an increased proportion of the chalcone glycoside isomers, the relative amounts of which depend upon the extent and duration of heating. Under our HPLC conditions which were chosen to separate the chalcone from flavylium form as rapidly as possible, the chalcone isomers probably would not separate but be collected as a single peak. The observed initial delay in formation of the flavylium form, referred to above (Fig. 4), could thus be due to photoisomerisation of *trans* chalcone to *cis* chalcone in the spectrophotometer, the exact rate of formation of the cationic anthocyanin depending on the proportion of

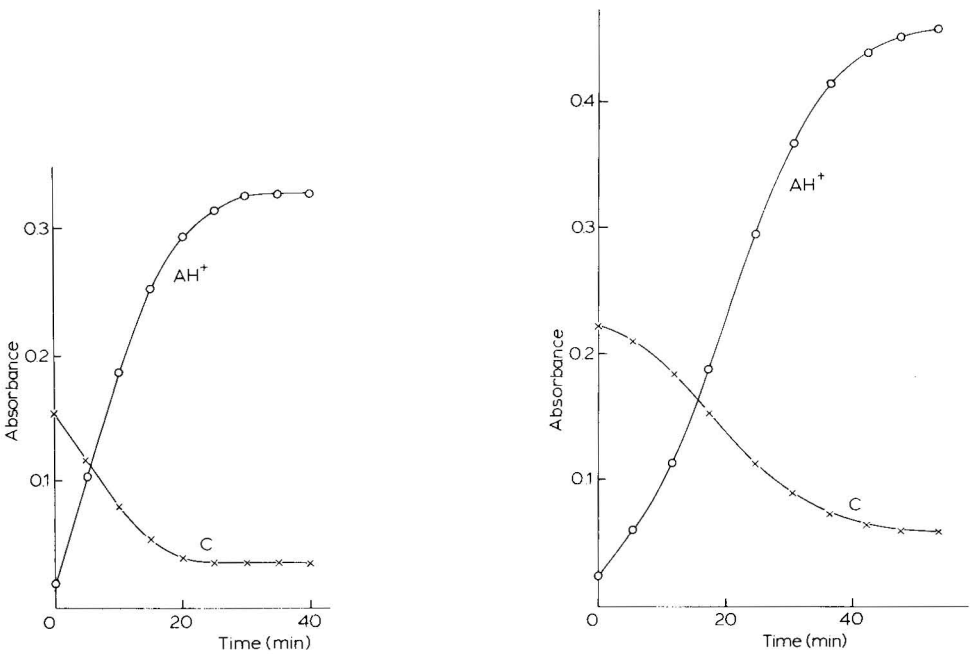

Fig. 3. Variation in the absorbance at the $\lambda_{max.}$ of the chalcone (C, 340 nm) and flavylium cation (AH^+, 524 nm) of malvidin 3-glucoside (solvent 75% A, 25% B). Solution exposed to light only during brief measurements at these wavelengths.

Fig. 4. Variation in the absorbance at the $\lambda_{max.}$ of the chalcone (C, 335 nm) and flavylium cation (AH^+, 522 nm) of malvidin 3,5-diglucoside during the spectral scans illustrated in Fig. 2.

trans chalcone in the mixture. Jurd[6] has reported such a process for the formation of certain flavylium salts in acid solutions and has found that in the absence of light, isomerisation of the *trans* chalcone is acid-catalysed and much slower. Consequently, it seems probable that the overall formation rate of flavylium cations of anthocyanins in acid solutions of the corresponding chalcones isolated by HPLC, depends on the relative proportions of the chalcone isomers in the mixture and on the degree of their exposure to light subsequent to their isolation. In the absence of light, the cationic structure should be formed initially almost entirely from the *cis* chalcone, and its rate of formation should thus be almost constant in the early stages. Measurements of the formation rate of the cationic form of malvidin 3-glucoside made with minimum exposure of the sample to light and UV radiation showed this to be so (Fig. 3).

The conversion of the chalcone was accompanied by a gradual change of the $\lambda_{max.}$ of the peak in the region of chalcone absorbance from 340 to 345 nm for malvidin 3-glucoside and from 335 to 340 nm for malvidin 3,5-diglucoside. The minor peaks remaining at the longer wavelengths after completion of the conversion must be attributed to the flavylium cationic forms of these anthocyanins rather than to small amounts of unconverted *trans* chalcone, since the chalcone could not be detected by HPLC in an equilibrated anthocyanin solution at pH 1.5.

Well-defined spectral isosbestic points at 395 and 295 nm were observed during the conversion of malvidin 3,5-diglucoside to its cationic form (Fig. 2). On two occasions all the spectral scans passed through these points; on the third occasion, the initial scans 1 and 2 did not pass through these isosbestics. In this respect similar behaviour was observed with the malvidin 3-glucoside chalcone-flavylium cation conversion. Scans 3–8 invariably passed through isosbestic points at 398 and 299 nm but this was not always so with scans 1 and 2. The inconsistency of the spectral scans 1 and 2 is in accord with the proposed photoisomerisation of the chalcone, but the significance of the absence or presence of isosbestic points will not be pursued because of the complexity of the system and the conclusions drawn from a previous study[8].

It is likely that chalcones of other anthocyanins may be separated by HPLC in a similar way. Indeed, during HPLC of *V. vinifera* grape skin extracts we have observed in addition to the chalcone of malvidin 3-glucoside, two peaks which may represent the chalcones of malvidin 3-acetylglucose and malvidin 3-*p*-coumaroylglucoside, these being the other anthocyanins present in greatest amounts in the samples studied. The peaks were not evident at 525 nm but were characterised by absorbance at 340 nm greater than at 280 nm. Peak sizes were increased on heating. We suggest that these criteria may be useful for detecting anthocyanin chalcones in other systems.

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Note

Separation of hydrazine, monomethylhydrazine, 1,1-dimethylhydrazine and 1,2-dimethylhydrazine by high-performance liquid chromatography with electrochemical detection

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Hydrazine (HY) is an important precursor for many industrial organic syntheses[1,2]. In addition, HY as well as monomethylhydrazine (MMH) and 1,1-dimethylhydrazine (UDMH) have seen extensive use as rocket propellants and fuels[3]. These compounds and, in particular, the closely related 1,2-dimethylhydrazine (SDMH) have been shown to be carcinogenic in laboratory animals[3–5]. Because of their widespread use and potent biological activity, considerable effort has been devoted to the development of methods for the detection and quantitation of these compounds in the environment and in biological fluids[6–11]. For the most part, such methods usually involve derivatization with either *p*-dimethylaminobenzaldehyde[7], pentafluorobenzaldehyde[9] or salicylaldehyde[11] followed by colorimetric estimation[7], gas chromatography with an electron-capture detector[9] or high-performance liquid chromatography (HPLC) with UV detection[11], respectively. Methods dependent on coupling with aldehydes are limited in application to hydrazines with an unsubstituted $-NH_2$ group; compounds such as SDMH which do not form hydrazones cannot be estimated in this way.

In this communication, we describe the separation of HY, MMH, UDMH and SDMH by ion-exchange HPLC using an electrochemical detector with glassy carbon working and auxiliary electrodes. The hydrazines do not require derivatization, and urine or blood plasma samples suspected of containing these compounds may be analyzed directly without previous work-up.

EXPERIMENTAL

HY, MMH, UDMH and SDMH–2HCl were purchased from Aldrich (Milwaukee, WI, U.S.A.). The hydrazines were dissolved in distilled deionized water ($\geqslant 13.7$ MΩ resistivity) which had been flushed with nitrogen to prevent air oxidation. In the case of SDMH–2HCl, all concentrations were calculated as the free base.

Stainless-steel columns of Aminex A-5 sulfonic acid type cation-exchange resin in the sodium form, particle size 13 ± 2 μm (Bio-Rad Labs., Richmond, CA, U.S.A.) were slurry packed using the same buffer and flow-rate as those ultimately used in the analyses. Various preliminary runs were made with short (5 to 13 cm long, 4 mm I.D.) columns and sodium borate buffers ranging from 0.01 to 0.05 *M* and from pH 8.6 to

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9.2 to establish optimal conditions. On the basis of these preliminary runs, a 30 cm × 4 mm I.D. column eluted with 0.05 *M* sodium borate buffer, pH 8.9, at 1 ml/min was selected for the separations described here.

A Waters Assoc. Model M6000A HPLC pump with a Model U6K injector were used in conjunction with a Metrohm Model EA1096 electrochemical detector. The latter used glassy carbon indicating and working electrodes and a Ag/AgCl reference electrode. Dead volume of the detector was approximately 3–4 μl. A Metrohm E506 Polarecord was used to supply the polarizing voltage (+1.0 V) and to detect cell current. Since this instrument was designed principally for application in differential pulse polarography where the movement of the chart recorder paper is discontinuous and is synchronized to the dropping of mercury, serrated HPLC recordings were obtained in our application. This circumstance was found not to interfere significantly with the determination of elution volumes or peak heights.

For the determination of hydrazines in rat blood plasma and urine, male F344 rats, approximately 300 g body weight, were injected subcutaneously with 50 mg/kg of UDMH or with 100 mg/kg SDMH, neutralized to pH 6.4 in 0.7% sodium EDTA. After injection, the rats were placed in metabolism cages within a fume hood[12] and blood samples were obtained from the orbital sinuses under light ether anesthesia. The blood, collected with heparinized capillaries, was centrifuged, and 100 μl of the resulting plasma was submitted directly to HPLC. Urine was collected for a period of 24 h after dosing in containers thermoelectrically cooled to 0–4°C. Because of the high levels of hydrazines excreted in the urine, it was necessary to dilute the samples with deionized water prior to HPLC in order to bring the concentrations to within the bounds of the standard curve.

RESULTS AND DISCUSSION

Typical HPLC profiles of the four hydrazines at three different concentrations are shown in Fig. 1. It is evident that the order of elution from the ion-exchange column is in order of increasing pK_a values. These are: UDMH, 7.21; SDMH, 7.52; MMH, 7.87; and HY, 8.07. While MMH, SDMH and UDMH show well defined peaks at levels down to 17, 10 and 8 ng, respectively, the HY peak is obscured by noise at levels below approximately 80 ng.

A standard curve relating the peak current obtained with the amount of hydrazines injected is shown in Fig. 2. For UDMH, SDMH and MMH the peak current is a linear function of quantity up to approximately 250 ng, 600 ng and 1100 ng, respectively. For HY a hyperbolic rather than linear relationship between peak current and quantity was obtained in the range examined.

Profiles obtained from the direct HPLC of blood plasma and urine from rats given UDMH or SDMH subcutaneously are shown in Figs. 3 and 4, respectively. Interestingly, the profile of urine from the rat given SDMH shows a small peak, corresponding in elution volume to MMH, which may represent a metabolite. However, SDMH normally contains a small amount of MMH as an impurity and an alternative possibility is that the MMH impurity in the dose may be preferentially concentrated by the animal and excreted in the urine.

Blood plasma levels of UDMH and SDMH following subcutaneous administrations of 50 and 100 mg/kg, respectively, are shown in Fig. 5. Although large in-

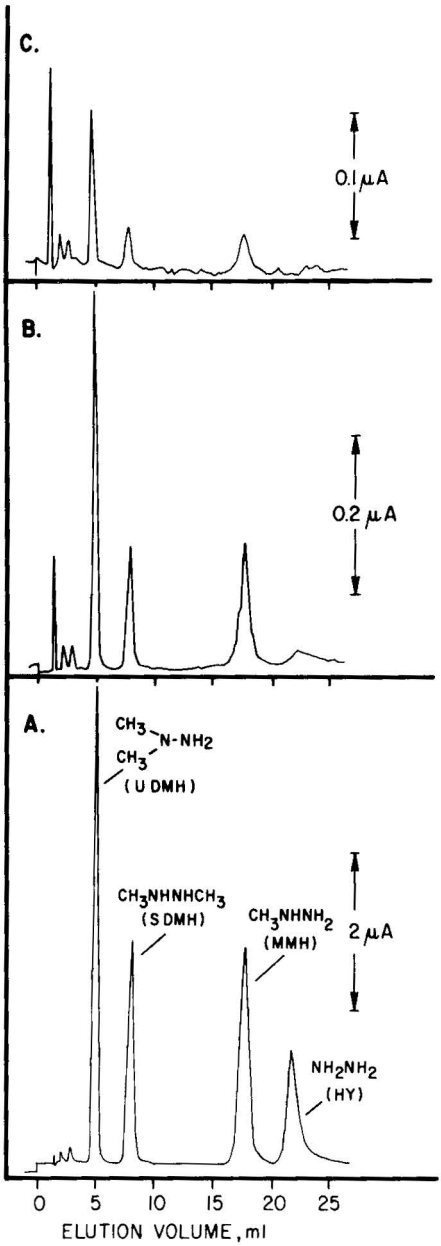
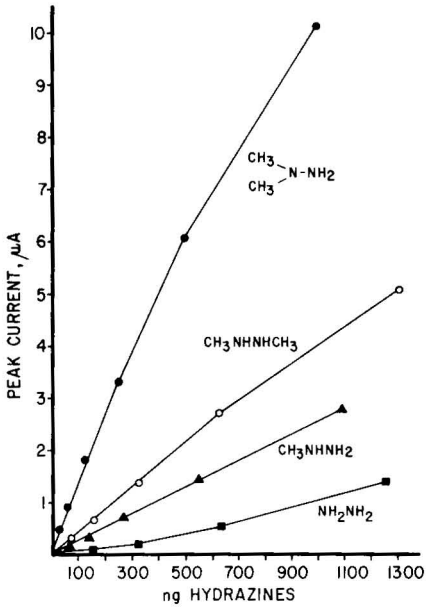


Fig. 1. HPLC of hydrazines on a 30 × 0.4 cm column of Aminex A-5 cation-exchange resin eluted with 0.05 *M* sodium borate, pH 8.90, at 1 ml/min. The amounts (in micrograms) of UDMH, SDMH, MMH and HY were, respectively, A: 0.5, 0.66, 1.09 and 1.26; B: 0.03, 0.04, 0.07 and 0.08; and C: 0.008, 0.01, 0.017 and 0.02.



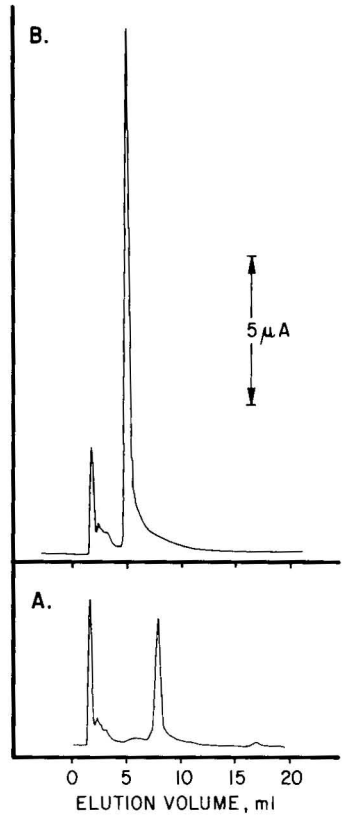
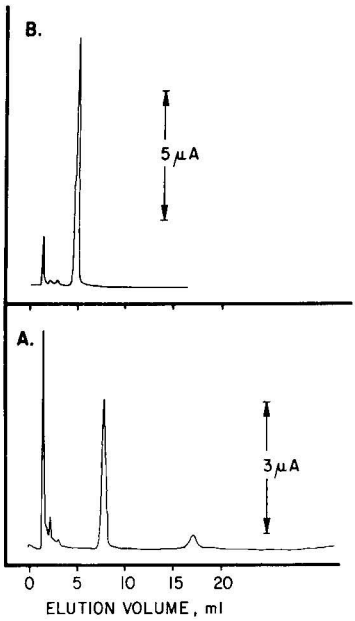


Fig. 2. Standard curve relating amounts of hydrazines injected and peak current. HPLC conditions same as for Fig. 1.

Fig. 3. HPLC of 100 μl of blood plasma obtained from a rat injected s.c. with 100 mg/kg SDMH (A) and with 50 mg/kg UDMH (B).

dividual variations in plasma levels of UDMH are apparent, nevertheless it is clearly evident that the plasma levels of UDMH are higher than those of SDMH at all but the earliest time points even though twice as much SDMH as UDMH was administered. The faster clearance of SDMH may be related to the metabolic conversion of the compound to azomethane gas[5,12] which is rapidly excreted via the exhaled air. Beyond 3 h, SDMH could not be detected in the blood, although UDMH was still detectable 6 hours after administration.

Previously, Rucki *et al.*[13] have reported the determination of procarbazine, a pharmacologically active hydrazine, by HPLC on amino-cyano columns with an amperometric detector with a carbon paste working electrode. The results of Rucki *et al.* together with ours demonstrate the utility of HPLC with electrochemical detection to the separation and estimation of various important hydrazine derivatives.



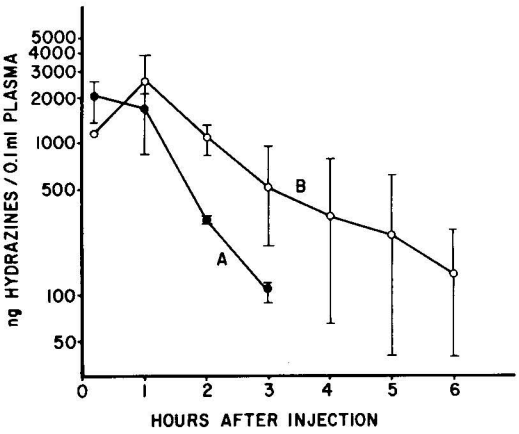


Fig. 4. HPLC of diluted urine samples obtained from a rat injected with 100 mg/kg SDMH (A) and with 50 mg/kg UDMH (B).

Fig. 5. Blood plasma levels of SDMH (A) and UDMH (B) in rats given these compounds as in Figs. 3 and 4. Data from three animals were averaged for each point. Brackets denote standard deviation.

ACKNOWLEDGEMENTS

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CHROM. 14,000

Note

High-performance liquid chromatographic separation of shallot volatile oil

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(Received May 20th, 1981)

The use of high-performance liquid chromatography (HPLC) for the separation of essential oils has already been tried on several occasions and has been discussed by Ross[1]. However, the column resolving power is often a limiting factor.

In the work reported here, a volatile oil from shallots was separated by HPLC and more than 90 peaks were detected by a UV detector. The resolution offered by HPLC is better than that offered by capillary column gas–liquid chromatography, which gives less than 90 peaks[2]. Six fractions obtained by HPLC separation can be identified directly by gas chromatography–mass spectrometry (GC–MS) without concentration; however, most other fractions have not yet been analysed.

EXPERIMENTAL

Shallots were purchased from the local market, chopped and steam-distilled by the AOAC method[3] to give volatile shallot oil.

HPLC was carried out with a Hewlett-Packard 1084B system with a 25 cm × 6.2 mm I.D. DuPont pre-packed Zorbax ODS (6 μm particles). Reagent-grade methanol was purchased from E. Merck (Darmstadt, G.F.R.), distilled using a 30-plate Kontes Oldershaw column and filtered through a 0.5-μm fluorocarbon filter (Millipore, Bedford, MA, U.S.A.). The methanol and Milli-Q deionized water (Millipore) were degassed for 30 min by stirring under vacuum before being used as eluents.

The shallot oil was diluted 50-fold with distilled methanol and filtered through a 0.2-μm fluorocarbon filter (Millipore), and 30 μl of the diluted shallot oil were applied to the HPLC system. Detection was effected with a Hewlett-Packard HP 79870A UV detector (254 nm). The solvent system was 62.5% B (methanol) in A (water) for the first 5 min, then a linear gradient programmed from 62.5% B to 90% B in the test 65 min, at a constant flow-rate of 2 ml/min.

Fractions were collected from each HPLC run and injected directly into a Hewlett-Packard 5985B GC–MS system equipped with a 74 cm × 2 mm I.D. glass column packed with 2% OV-101 + 0.2% Carbowax 20M on 100–120-mesh Chromosorb W HP. The operating conditions of mass spectrometer were ionization voltage 70 eV, source temperature 200°C and accelerating voltage 1800 V.

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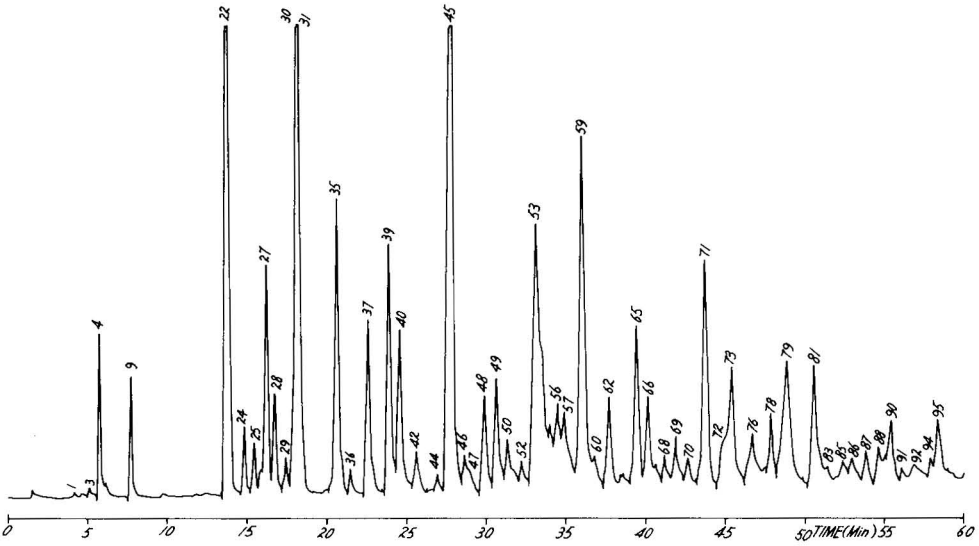

Fig. 1. High-performance liquid chromatogram of shallot oil.

RESULTS AND DISCUSSION

Fig. 1 shows the separation of shallot oil by HPLC. The integration of the peaks in Fig. 1 is shown in Table I. These data are valuable if HPLC is used for

TABLE I

PERCENTAGE OF EACH PEAK RELATIVE TO THE TOTAL PEAK AREA IN THE HIGH-PERFORMANCE LIQUID CHROMATOGRAM OF SHALLOT OIL

Percentages were calculated according to the absorbance of each peak at 254 nm. Peak Nos. ref to Fig. 1.

Peak No.	*Percentage*	*Peak No.*	*Percentage*
4	1.057	56	1.270
9	0.996	59, 60	6.684
22	9.895	62	1.304
24	0.754	65	2.381
25	0.622	66	1.372
27	2.828	68	0.254
28	1.277	69	0.665
29	0.488	70	0.331
30, 31	10.623	71	3.690
35	4.110	73, 74	3.116
36	0.294	78	0.750
37	2.531	79	3.221
39	3.667	81	2.424
40	2.586	85	0.122
42	0.785	86	0.198
44	0.113	87	0.407
45	12.480	88	0.299
48	1.475	90	0.552
49	1.680	95	0.906
53, 54	6.612		

TABLE II

IDENTIFICATION OF COMPOUNDS IN SHALLOT OIL

Peak Nos. refer to Fig. 1.

Peak No.	*Compound*
22	Dimethyl trisulphide
37	1-Methylthiopropyl ethyl disulphide
45	Methyl propyl trisulphide
53	Dipropyl trisulphide
59	Propyl propenyl trisulphide
71	Dipropyl trisulphide

quality evaluation. Liquid chromatography is the method of choice in the evaluation of essential oils, as they may be sensitive to temperature.

Table II shows the fractions from HPLC identified directly by GC–MS analysis. The other minor fractions cannot be examined owing to the low solute concentration. During the past year considerable progress has been made with combined liquid chromatography–mass spectrometry[4,5]. However, the ability to combine reversed-phase liquid chromatography with on-line mass spectrometry, without seriously compromising either technique, is still a difficult problem. In this paper, a reversed-phase LC–GC–MS technique is proposed.

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Note

Analysis of 2-hydroxybenzophenone and 2′-hydroxyphenylbenzotriazole UV stabilizers by high-performance liquid chromatography

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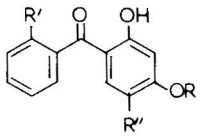
(Received May 26th, 1981)

Substituted 2-hydroxybenzophenones (I) and 2′-hydroxy-2-phenylbenzotriazoles (II) are widely used as light stabilizers in polymeric materials to protect the substrate from photo-oxidation by sunlight. In the course of our studies of the reactions of these stabilizers during the autoxidation and photo-oxidation of polymers and model systems it was necessary to determine quantitatively the concentration remaining in the oxidized substrate. In this note a procedure is described for the determination of 2-hydroxybenzophenone and 2′-hydroxy-2-phenylbenzotriazole light stabilizers by high-performance liquid chromatography (HPLC) on silica gel.



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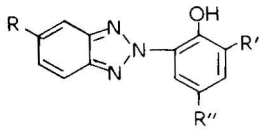

II

EXPERIMENTAL

Materials

2-Hydroxybenzophenone (Aldrich, Milwaukee, WI, U.S.A.) was purified by column chromatography. 2′-Hydroxy-2-phenylbenzotriazole (Ciba-Geigy, Basle, Switzerland) and 2-hydroxybenzophenone (Cyanamid, Bound Brook, NJ, U.S.A.) light stabilizers, shown in Table I, were recrystallized before use. Reagent-grade methylene chloride was purified by passing through activated (Gr. 1) silica gel and distilled. Isopropanol was spectroscopic grade.

HPLC

HPLC separations were carried out on an isocratic system (Altex 110A pump, Rheodyne loop injection valve with 10-μl loop and Altex 153 UV detector). A 15 cm $\times$ 4.6 mm silica gel column (5 μm Spherisorb) and 5 cm pre-column (slurry packed with 5 μm Spherisorb) were used for separations. The mobile phase was 0.1% iso-

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TABLE I

STRUCTURES OF THE PHENOLIC LIGHT STABILIZERS

Additive	*Substituents*		
	R	*R'*	*R''*
2-Hydroxybenzophenones (I)			
2,4-Dihydroxybenzophenone	H	H	H
Cyasorb UV 9	CH_3	H	H
Cyasorb UV 531	n-C_8H_{17}	H	H
Cyasorb UV 24	CH_3	OH	H
Cyasorb UV 207	CH_3	CO_2H	H
Cyasorb UV 284	CH_3	H	SO_3H
2'-Hydroxy-2-phenylbenzotriazoles (II)			
Tinuvin P	H	H	CH_3
Tinuvin 320	H	$C(CH_3)_3$	$C(CH_3)_3$
Tinuvin 326	Cl	$C(CH_3)_3$	CH_3
Tinuvin 327	Cl	$C(CH_3)_3$	$C(CH_3)_3$
Tinuvin 328	H	$C(CH_3)_2CH_2CH_3$	$C(CH_3)_2CH_2CH_3$

propanol in methylene chloride at a flow-rate of 1.0 ml/min. The column was activated by pumping dry diethyl ether (dried with 4A molecular sieve) and the activity maintained for longer periods by inclusion of a 15-cm column packed with a coarser grade of silica gel before the injector.

Derivatization

Approximately 0.5 mg of the phenolic light stabilizer was acetylated by addition of 1.0 ml of a solution of acetic anhydride (2.0 ml) and triethylamine (4.0 ml) in methylene chloride (10.0 ml). The hydroxybenzophenones and Tinuvin P were allowed to stand for 1 h at room temperature whilst Tinuvins 320, 326, 327 and 328 were heated under gentle reflux for 2 h. The solution was then evaporated to dryness under reduced pressure and made up to 5.0 ml with methylene chloride for analysis.

RESULTS AND DISCUSSION

HPLC separation of phenolic antioxidants on silica gel has been applied to the analysis of phenolic antioxidants and their transformation products in polymers[1–3]. However, chromatographic separation of the 2-hydroxybenzophenone and 2'-hydroxy-2-phenylbenzotriazole light stabilizers on silica gel results in broad tailing peaks (Fig. 1) the retention times and peak shapes of which are strongly dependent on the degree of activation of the silica gel and the presence of polar modifiers in the mobile phase. This behaviour is presumably due to strong hydrogen-bonding interaction between the surface silanol groups and the stabilizer molecules (III and IV). (2-Hydroxybenzophenones are known to form strong intramolecular hydrogen bonds[4]). HPLC analysis of these polymer additives would therefore be expected to be improved by suitable derivatization of the phenolic hydroxyl group.

Attempts to methylate the 2-hydroxy group of the 2-hydroxybenzophenones

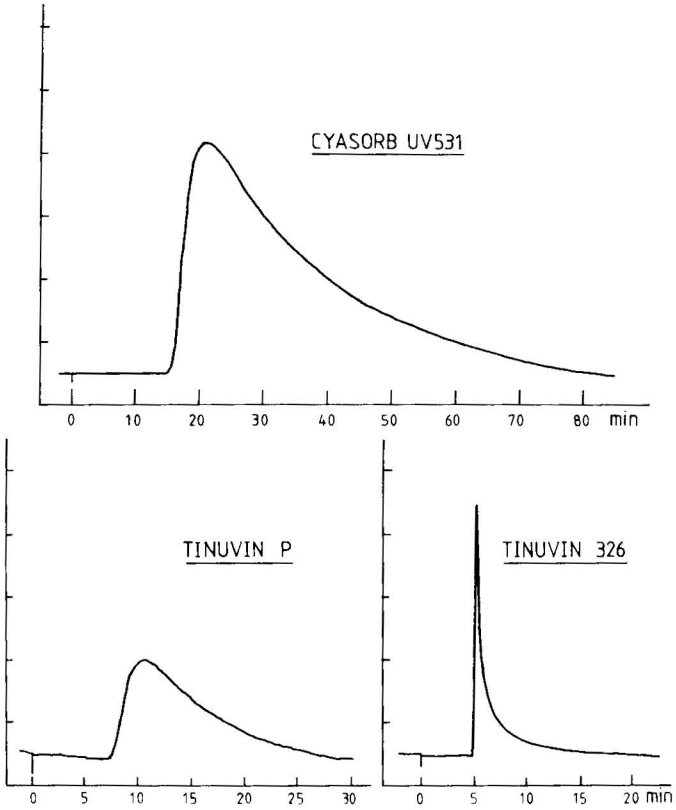
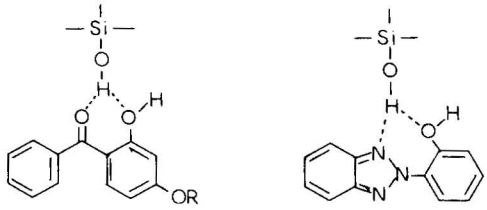


Fig. 1. Chromatographic behaviour of Cyasorb UV 531, Tinuvin P and Tinuvin 326 on silica gel. Mobile phase: 0.1% isopropanol in methylene chloride, 1.0 ml/min.



III IV

with diazomethane and diazomethane–boron trifluoride, in ether proved unsuccessful. In each case thin-layer chromatography showed that starting material was recovered unchanged, except for 2,4-dihydroxybenzophenone where 2-hydroxy-4-methoxybenzophenone was obtained on treatment with diazomethane.

Acetylation was more successful and the corresponding acetates chromatographed on silica gel with greatly improved peak shape and reproducibility of retention times. A wide range of retention times was obtained for the commercial additives studied enabling their identification and analysis by this method.

Acetylation of the phenolic light stabilizers was performed under mild conditions using acetic anhydride and triethylamine in methylene chloride. For the hydroxybenzophenones and the unhindered benzotriazole, Tinuvin P (Table I), acetylation was carried out at room temperature and was complete, as verified by HPLC, in less than 1 h. However, for the more hindered benzotriazoles Tinuvins 320, 326, 327 and 328 (see Table I), with a tertiary alkyl group *ortho* to the phenolic hydroxyl group, acetylation was slower and complete reaction, as verified by HPLC, required heating at the boiling point of methylene chloride (40°C).

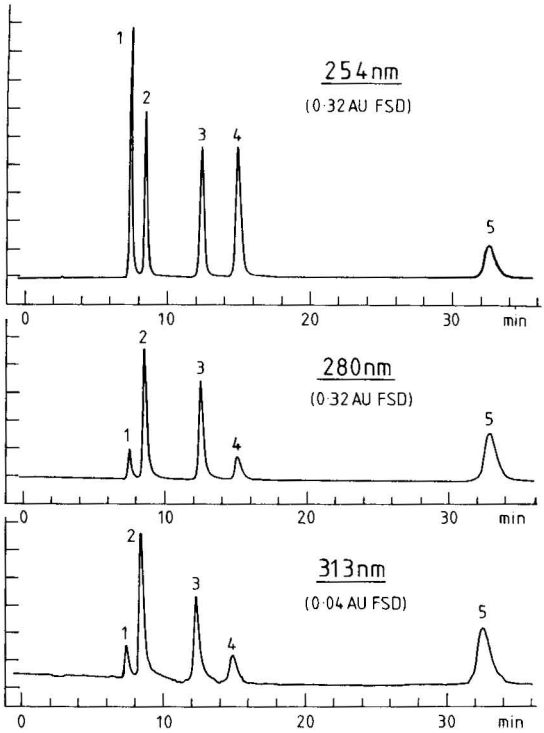


Fig. 2. HPLC separation of 2-hydroxybenzophenone light stabilizers on silica gel after acetylation. Peaks: 1 = 2-hydroxybenzophenone; 2 = Cyasorb UV 531; 3 = Cyasorb UV 9; 4 = 2,4-dihydroxybenzophenone; 5 = Cyasorb UV 24. Column, Spherisorb 5 μm; mobile phase 0.1% isopropanol in methylene chloride, 1.0 ml/min.

Fig. 2 shows the HPLC separation of several commercial 2-hydroxybenzophenone light stabilizers and 2-hydroxybenzophenone on silica gel after acetylation Cyasorb UV 207 (not shown in Fig. 2) gave a retention time of 6.2 min whilst Cyasorb UV 284 (2-hydroxy-4-methoxy-5-sulphobenzophenone) was not eluted under the conditions employed.

Fig. 3 shows the separation of several commercial 2′-hydroxy-2-phenylbenzotriazole light stabilizers after acetylation. Retention times for the acetates of these additives are shorter than those of the hydroxybenzophenones although the stabilizers are still well resolved. Figs. 2 and 3 show the chromatographic traces at three detector wavelengths (254, 280 and 313 nm). The different spectral

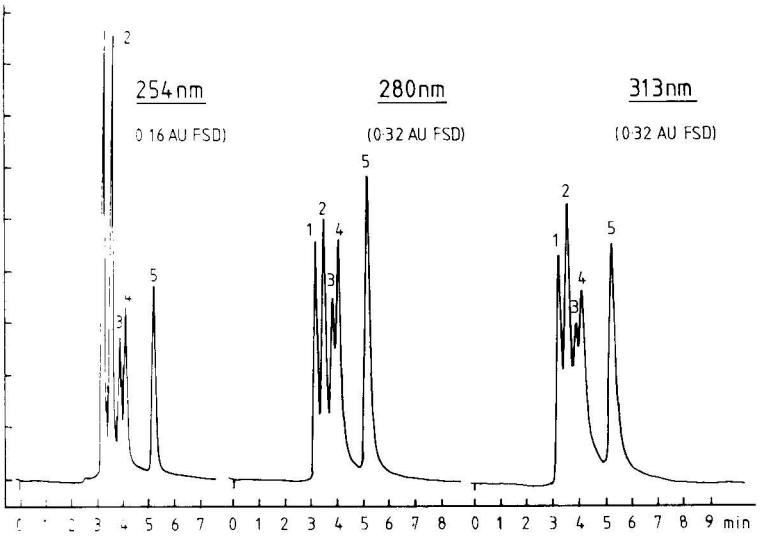


Fig. 3. HPLC separation of 2'-hydroxy-2-phenylbenzotriazole light stabilizers on silica gel after acetylation. Peaks: 1 = Tinuvin 327; 2 = Tinuvin 326; 3 = Tinuvin 328; 4 = Tinuvin 320; 5 = Tinuvin P. Conditions as in Fig. 2.

properties of the individual stabilizer derivatives aid in the characterization of the polymer additive.

The application of the above HPLC method to the determination of extraction yields of hydroxybenzophenone and hydroxyphenylbenzotriazole light stabilizers from polypropylene film is shown in Fig. 4. The extraction of 170 μm melt-pressed

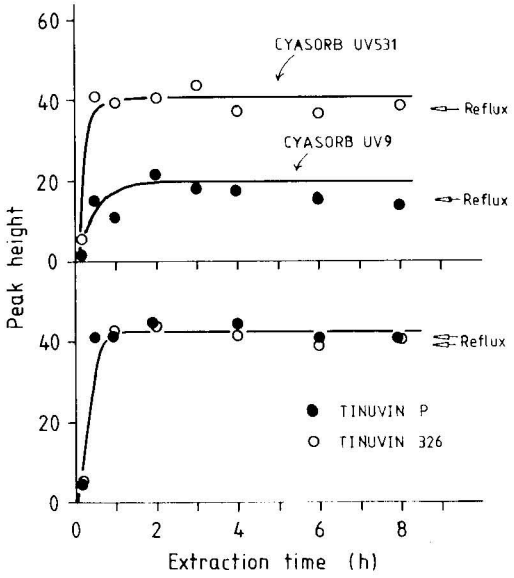


Fig. 4. Extraction yields of phenolic light stabilizers from 170 μm polypropylene films with methylene chloride at room temperature.

films containing 0.5% (w/w) of the additive was carried out by shaking with methylene chloride at a temperature of *ca.* 25°C. The figure shows that the additives were completely extracted from the film in less than 2 h. The same final extraction yields were obtained on refluxing the film sample in methylene chloride for 8 h indicating the complete removal of the additive from the film in the room temperature extractions.

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Note

Quantitation of cocaine in a variety of matrices by high-performance liquid chromatography

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(Received May 29th, 1981)

In forensic science laboratories cocaine is rarely encountered as a pure drug. Typically lignocaine and procaine are often added as adulterants, and clandestinely prepared cocaine from "Coca" leaves contains very small quantities of cinnamoyl cocaine. Gas chromatography (GC) can be used for the separation of cocaine from the cinnamoyl cocaine isomers[1], and other synthetic caines provided that there is sufficient material available. Since this method lacks the sensitivity to detect the small quantities of cinnamoyl cocaine present in clandestinely prepared samples of cocaine, the technique is not entirely suitable for confirming the origin of the cocaine. Thin-layer chromatography (TLC) can be used for qualitative analysis of cocaine[2], and UV spectroscopy can be employed to quantitate cocaine provided that there is no cinnamoyl cocaine present. Cinnamoyl cocaine has a very high $E^1_{1\%}$; (360 at 270 nm) in comparison with cocaine ($E^1_{1\%}$; 36 at 270 nm) and therefore traces of the cinnamoyl cocaine can give false quantitative results by UV. The high-performance liquid chromatographic (HPLC) method reported in this paper provides accurate quantitation of cocaine in any matrix and allows discrimination between clandestinely and synthetically prepared samples of cocaine. Both diastereomeric forms of cinnamoyl cocaine were detected and separated by this method of analysis. Methods of preparation of cocaine analogues for use as a reference materials are also reported.

EXPERIMENTAL

High-performance liquid chromatography

Separations were performed on a 15 cm × 4.6 mm I.D. stainless-steel column packed with LiChrosorb RP-2 packing material (5 μm; E. Merck, Darmstadt, G.F.R.). Methanol–0.1 *M* ammonium nitrate (40:60) adjusted to pH 4.3 with 2 *M* hydrochloric acid was pumped through the column at 1.5 ml min^{-1}, and the eluent was monitored at 279 nm with a variable wavelength UV detector (CE 202; Cecil Instruments, Cambridge, Great Britain). Samples were dissolved in the eluent and injected onto the top of the column via a stop-flow injection system[3].

Gas chromatography–mass spectrometry (GC–MS)

A Varian 1400 gas chromatograph (Varian, Palo Alto, CA, U.S.A.) fitted with a 3 ft. × 4 mm I.D. glass column containing 3% OV-17 on Gas-Chrom Q (100–120

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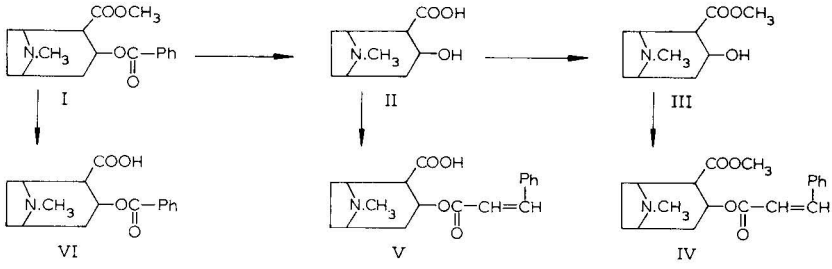
mesh) packing material was linked to a VG Micromass 12-12F quadropole mass spectrometer (VG Analytical, Cheshire, Great Britain). For electron impact studies the separator was maintained at 230°C and the ion source at 200°C and 70 eV. For the chemical ionisation studies isobutane was introduced into the source which was maintained at 170°C and 50 eV with an emission current of 250 μA.

Thin-layer chromatography

TLC was carried out on a 10 × 5 cm plate coated with silica gel 60 F_{254} (E. Merck), and treated with sodium hydroxide. The plates were developed with methanol–acetone (3:1), and the compounds were identified initially by UV adsorption at 254 nm and then by spraying with acidified iodoplatinate solution.

Preparation of compounds related to cocaine

Reference materials were required for this study, and they were prepared as shown below. Ecgonine (II), methyl ecgonine (III), cinnamoyl cocaine (IV), cinnamoyl ecgonine (V) and benzoyl ecgonine (VI) were prepared either directly or indirectly from cocaine (I).



Ecgonine (II)

According to the method of Bell and Archer[4], cocaine (5.5 g) was treated with aqueous hydrochloric acid (130 ml; prepared by adding 10 ml concentrated acid to 150 ml water). The solution was refluxed for 15 h and then extracted several times with ether. The aqueous phase was evaporated to dryness to give a white solid which was recrystallised from ethanol to yield white crystals of ecgonine hydrochloride.

Cinnamoyl cocaine (IV)

Ecgonine hydrochloride (486 mg) in methanol (3 ml) was treated with a solution of diazomethane in ether (5 ml). After 0.5 h at room temperature all the starting material had dissolved. TLC showed a single spot due to methyl ecgonine (III). The solution was evaporated, dissolved in benzene (40 ml) and refluxed for 6 h with cinnamic anhydride (*trans* isomer; 1.83 g) and sodium carbonate (300 mg). The mixture was cooled, filtered and the filtrate was washed with dilute hydrochloric acid (0.5 *M*; 3 × 20 ml). The aqueous phase was basified with ammonia (sp. gr. 0.88) and extracted with ether (3 × 10 ml). The combined ethereal extract was washed with hydrochloric acid (0.5 *M*; 2 × 5 ml). The aqueous phase was saturated with sodium carbonate and a white solid was precipitated. The solid was removed by filtration, dried and recrystallised from light petroleum (b.p. 60–80°C) to give white crystals of cinnamoyl cocaine.

Cinnamoyl ecgonine (V)

Ecgonine hydrochloride (222 mg), cinnamic anhydride (417 mg), benzene (5 ml) and sodium carbonate (100 mg) were refluxed for 2.5 h. After cooling the reaction mixture was filtered and the filtrate was evaporated to give a viscous gum. This was stirred with ether and the ethereal solution was removed and evaporated. The residue obtained was dissolved in ethanol and a small quantity of ether was added, and on standing crystals were deposited. These were filtered off and dried to give cinnamoyl ecgonine.

Benzoyl ecgonine (VI)

Cocaine (250 mg) was refluxed with water (15 ml) for 3 h. The solution was extracted with ether and the aqueous phase was evaporated to dryness. The solid was recrystallised from water to give white crystals of benzoyl ecgonine.

Structures and molecular weights of the prepared compounds were confirmed by GC–MS, and purities were established by GC, HPLC and TLC.

By comparison with the infrared spectra and GC data reported for cinnamoyl cocaine isomers[1], the compound prepared by the above route gave the trans isomer. No cis isomer could be detected by GC or HPLC.

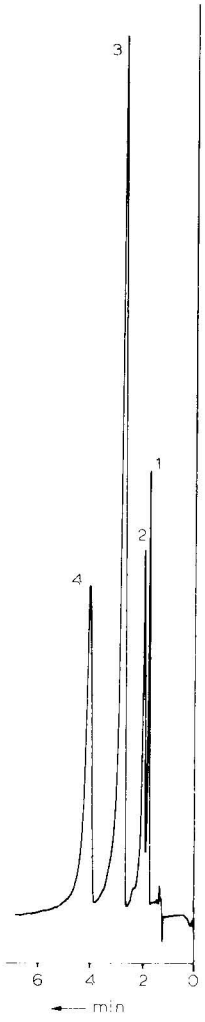
RESULTS AND DISCUSSION

In agreement with several workers[5,6] it was found that weak protolytes *i.e.*, cocaine, were difficult to chromatograph on silica or octadecyl modified silica with aqueous methanol eluents, because their amino functional groups are easily protonated. With both packing materials, methanol concentration, ionic strength and pH affected peak shape and retention time and no suitable combination could be obtained to effect a satisfactory result for the compounds that were to be separated. Following further investigations good chromatographic separations and peak profiles were eventually obtained on a reversed-phase LiChrosorb RP-2 packing material. The separation mechanism for this system is complex, but it is believed that both partition with the bonded portion of the packing material and adsorption on the unreacted surface silanol groups are responsible for the separations. Using the conditions described in the experimental section the separation obtained for a standard containing procaine, lignocaine, cocaine and cinnamoyl cocaine is shown in Fig. 1.

With exception of cinnamoyl cocaine, standards were stable in eluent for several weeks. Cinnamoyl cocaine was found to decompose in this solvent after a few days, but no decomposition was detected when this material was stored in chloroform.

Retention data for the cocaine analogues, synthetic caines and some other drugs that have been detected in cocaine samples are shown in Table I.

Since cinnamoyl cocaine has a relatively high $E^{1}_{1\%}$ in comparison with cocaine the very low levels of the former compound were easily detected with the UV detector. Discrimination between clandestinely prepared and synthetically produced cocaine was possible because only the clandestinely prepared material contains cinnamoyl cocaine. Of the 336 cocaine samples that were analysed during the last year in this laboratory, 126 were found to contain cinnamoyl cocaine. Lignocaine, procaine and benzocaine were found in 60 of these samples, and mannitol, glucose and lactose were



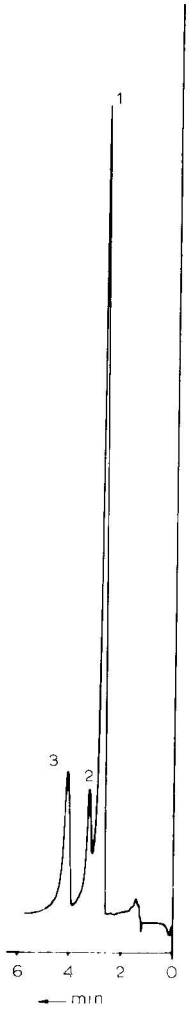


Fig. 1. Chromatogram of a standard mixture. Peaks: 1 = procaine; 2 = lignocaine; 3 = cocaine; 4 = *trans*-cinnamoyl cocaine. Conditions of analysis are as stated in the text.

Fig. 2. Chromatogram showing the components that are commonly detected in clandestinely prepared samples of cocaine. Peaks: 1 = cocaine; 3 = *trans*-cinnamoyl cocaine. Peak 2 postulated as *cis*-cinnamoyl cocaine following preparative HPLC and GC–MS studies. Conditions of analysis are as stated in the text.

detected as diluents in 70 samples. Several samples were found to contain as many as four drugs in combination with cocaine.

In a large number of the clandestinely prepared cocaine samples, another component was detected with an relative retention time of 1.18, and a typical example is shown in Fig. 2.

To help in the characterisation of this material preparative HPLC was employed. By increasing the ionic strength of the aqueous phase to 0.2 *M* ammonium nitrate the component was nearly resolved from the cocaine and fractions were col-

TABLE I

HPLC RETENTION DATA

Relative retention times (RRT values) were calculated with respect to cocaine (retention time 2.7 min)

Compound	*RRT*	*Compound*	*RRT*
Cocaine analogues and synthetic caines		*Alkaloids*	
Procaine	0.65	Morphine	0.45
Chloroprocaine	0.67	Codeine	0.52
Lignocaine	0.70	Monoacetylmorphine	0.61
Pyrrocaine	0.74	Diamorphine	0.91
Benzoyl Ecgonine	0.77	Acetylcodeine	0.92
Dimethocaine	0.77		
Octacaine	0.77	*Basic drugs*	
Propoxycaine	0.80	Ephedrine	0.58
Prilocaine	0.83	Caffeine	0.60
Mepivacaine	0.83	Amphetamine	0.69
Orthocaine	0.89	Methylamphetamine	0.69
Cocaine	1.00	Cyclizine	2.58
Benzocaine	1.13	Dipipanone	2.58
Butanilicaine	1.13		
Piperocaine	1.17		
cis-Cinnamoyl cocaine	1.18		
Leucinocaine	1.26		
Proxymetacaine	1.32		
Amylocaine	1.41		
Butacaine	1.47		
trans-Cinnamoyl cocaine	1.49		
Amydricaine	1.69		
Phenacaine	1.72		
Cinchocaine	3.00		
Cyclomethycaine	3.00		

lected, pooled, extracted and analysed by GC–MS. The spectrum obtained was identical to that of our *trans*-cinnamoyl cocaine, but the retention times were shorter for this unknown by both GC and HPLC. It was postulated that this compound was the *cis*-isomer of cinnamoyl cocaine.

With the analytical conditions described earlier the lower limit of detection was 100 ng for cocaine, and the UV detector response was found to be linear over the range 0.01 to 0.2 a.u.f.s. A relative standard deviation of less than 3% was obtained from peak height measurements for a cocaine standard, and quantitation of cocaine in a sample was therefore obtained by calculating the sample to standard peak height ratio without the use of an internal standard.

Reproducibility of this method of quantitation was established by analysing ten accurately weighed samples of a mixture which contained cocaine. Duplicate injections of these samples were followed by an injection of a cocaine standard, and the purity of each sample was determined by comparing peak heights with that of the standards. A relative standard deviation of 5% was recorded and therefore this method of quantitation was shown to be both reproducible and rapid.

CONCLUSIONS

Using HPLC with UV detection and a LiChrosorb RP-2 reverse phase packing material cocaine could be detected in a variety of matrices. The ability of the system to separate cocaine from cinnamoyl cocaine enables accurate quantitation of cocaine. A method of comparing the peak heights of cocaine in samples against a cocaine standard of known concentration gives rapid and reproducible results.

The detection of cinnamoyl cocaine in samples can be used as a method of discrimination, since only clandestinely prepared cocaine will contain this material. Some samples of clandestinely prepared cocaine were found to contain both isomeric forms of cinnamoyl cocaine which could be easily separated by this method of analysis.

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Note

Semi-preparative high-performance liquid chromatography and spectroscopic characterisation of eight geometric isomers of leukotriene A methyl ester

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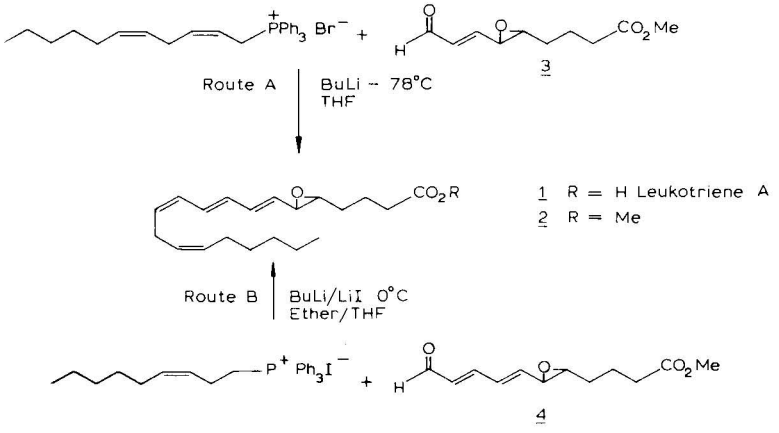
Leukotriene A (1) is now recognised as the precursor of leukotriene B, a compound with potent chemotactic activity for polymorphs, and leukotrienes C and D. The latter compounds have recently been shown[1] to be related to SRS-A which is thought to be the main mediator in human asthma. The biological potency of these compounds is resulting in intense synthetic activity in this new pathway of arachidonic acid metabolism[2,3]. The stereochemistry of these molecules must be characterised to enable satisfactory comparison of natural and synthetic compounds, as well as explore their structure–activity relationships.

The synthesis initially used to prepare the methyl ester of leukotriene A (2), using a 9 carbon epoxide (3) has been described previously[4–6] (route A). A route similar to that described by Corey[6], utilizing an 11 carbon epoxide (4), was also used (route B). Both reactions have yielded a number of isomers of 2.



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Due to the complexity of the reaction product, a high-performance liquid chromatographic (HPLC) separation and mass spectrometry (MS) and ultraviolet (UV) characterisation of the eluate peaks was required, prior to stereochemical assignments.

This paper describes the chromatographic separation and MS and UV characterisation of eight geometric isomers of 2 and an isomeric tetraene molecule (5).

EXPERIMENTAL

Reagents

The Spherisorb S5W and S5NH column packing material was supplied by Phase Separations (Queensferry, Great Britain). HPLC grade hexane was purchased from Fisons (Loughborough, Great Britain), analytical-reagent grade diethyl ether from May and Baker (Dagenham, Great Britain) and analytical-reagent triethylamine from BDH (Poole, Great Britain).

Instrumentation

Chromatography was performed with a constant-flow Milton Roy constametric IIG pump and a Cecil 212 variable wavelength UV detector set at 276 nm for analytical separations and 296 nm to reduce sensitivity for preparative work. The detector was fitted with a 10-mm path length flow-cell for analytical chromatography and a 1-mm path length flow-cell for preparative chromatography. Samples were injected using a Rheodyne variable volume valve injector fitted with a 20-μl loop for analytical separations, and a 2.0-ml loop for preparative separations.

Chromatography

All stainless-steel columns were packed in a vertically upwards mode from a methanol slurry of the packing material. Analytical chromatography was performed on 12.5 cm × 5 mm I.D. columns packed with either Spherisorb S5W or S5NH material. The isomers were eluted with hexane–diethyl ether–triethylamine (95:5:0.5) at a flow-rate of 1.0 ml/min. Preparative separations were carried out on 50 cm × 8 mm I.D. columns packed with the same materials. The identical eluting solvent was used at a flow-rate of 5.0 ml/min.

Mass spectrometry

The required HPLC eluate was reduced in volume before transference to a sample holder tube and the sample blown to dryness. All spectra were recorded by an LKB 9000S mass spectrometer. Samples were analysed by direct insertion probe at 50°C using an ion accelerating voltage of 3.5 kV, an electron voltage of 20 eV and a source temperature of 270°C.

UV spectrometry

The UV absorption spectra were recorded in cyclohexane on a Pye Unicam SP8-100 spectrophotometer calibrated at 279.4 nm with a Holmium filter.

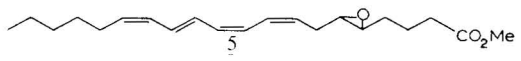
RESULTS AND DISCUSSION

Previous workers[4–6] have reported the presence of geometric isomers of the methyl ester of leukotriene A (2) associated with the stereochemistry of the 9,10- and 11,12-double bonds. As these molecules had similar R_F values in a number of thin-layer chromatography solvent systems[4], or were not considered chromatographically separable[6], no attempt was made to separate the molecules using preparative chromatographic techniques. We initially observed the presence of only three of the four potential geometric isomers[7] associated with the 9,10- and 11,12-double bonds in these leukotrienes. However, more recent reaction mixtures obtained under modified experimental conditions have shown, in addition to the presence of the fourth geometric isomer of 2, a further four minor isomers.

Chromatography

An optimum HPLC separation was obtained using a Spherisorb S5W column and an ether–hexane eluent. Triethylamine was incorporated in the solvent system as leukotriene A methyl ester is unstable under acidic conditions. Although the crude reaction mixtures (route A) were initially purified on a gravity-feed silica column, they still contained over ten components. Four main components isolated by HPLC were shown by MS and UV analysis to be isomers of leukotriene A methyl ester.

As the isomers were only just resolved under analytical conditions, a high-performance semi-preparative column was required to separate the required amounts. The use of a semi-preparative S5W column, which had an efficiency of over 50,000 theoretical plates for a *m*-nitroaniline standard, allowed the separation of up to 3 mg of crude material per injection. Isomers 2a, 2b and 2d appeared chromatographically pure on re-analysis. Isomer 2c showed a 7% impurity, identified as the tetraene (5). As these latter two compounds are chromatographically well resolved, it was assumed that 5 was formed as a rearrangement[8] product from 2c.



5

In samples obtained from route B, a minor peak eluted before isomer 2a on the semi-preparative column. This component was isolated and appeared by MS and UV analysis to be a fifth isomer (2e) of leukotriene A methyl ester.

In certain samples the chromatographic peak assigned as isomer 2a (route A and route B) was seen to have a slight shoulder on the tail of the peak. Further analytical HPLC on an S5NH column of selected semi-preparative fractions of 2a showed the presence of a further component. Semi-preparative HPLC of these frac-

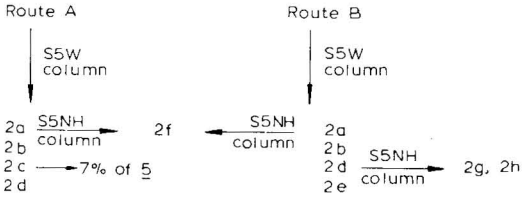


Fig. 1. Purification route used for isomers 2a–2h.

tions on an S5NH column (40,000 theoretical plates for a *m*-nitroaniline standard) and subsequent MS and UV analysis showed the presence of a sixth isomer (2f). Isomers 2b–2e were also examined on a Spherisorb S5NH column. No further impurities were observed in isomers 2b, 2c and 2e although isomer 2d was found to contain two impurities. Isomer 2d was therefore further purified on the semi-preparative S5NH column. The two impurities were isolated and were shown by MS and UV analysis to be further isomers (2g and 2h) of leukotriene A methyl ester.

In summary, a total of eight geometric isomers of leukotriene A methyl ester were isolated using a combination of the semi-preparative Spherisorb S5W and S5NH columns. The purification route used for each isomer is shown in Fig. 1. A separation of isomers 2a-2e and 5 on the S5W and S5NH columns is shown in Fig. 2a and 2b, respectively. The minor isomers 2g and 2h are not observed under either of these conditions as they coelute with the major isomers. To obtain isomers 2f–2h it was necessary to isolate the fractions corresponding to 2a and 2d on the S5W column and reprocess these fractions on the S5NH column. A comparison of the separations of the major isomers 2a and 2d and minor isomers 2f, 2g and 2h on a semi-preparative S5W and S5NH column is shown in Fig. 2c and 2d, respectively.

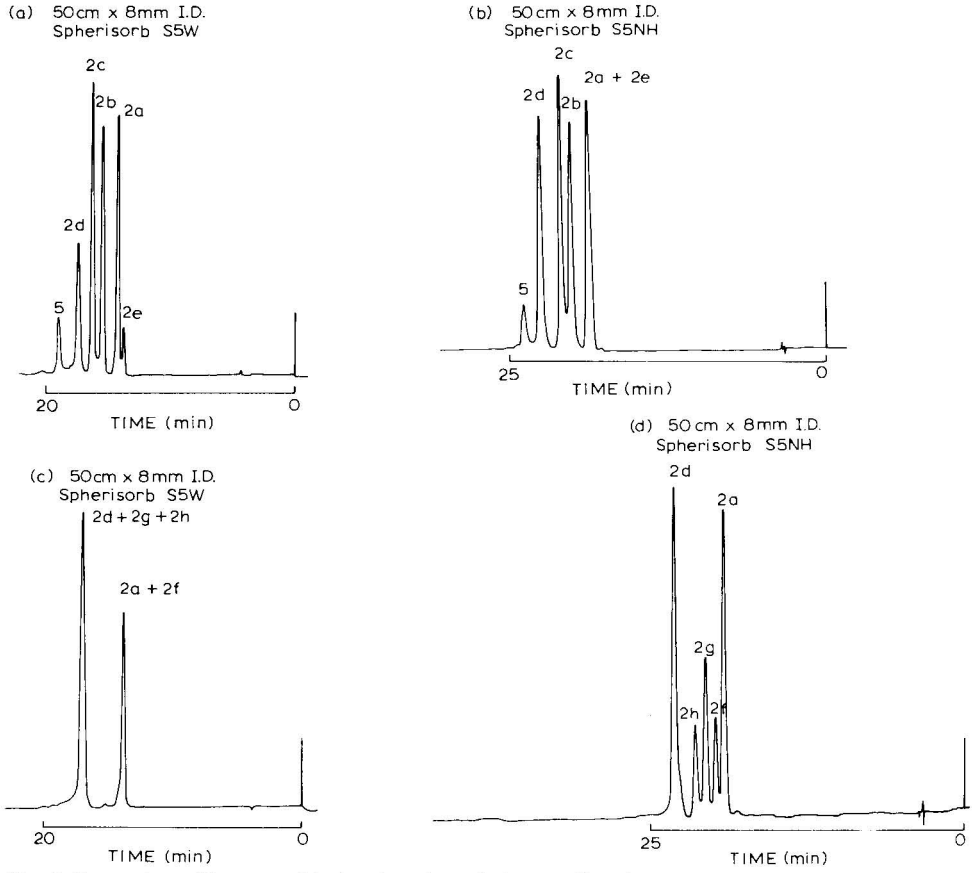


Fig. 2. Separation of isomers of leukotriene A methyl ester (2) and tetraene (5) on Spherisorb S5W and S5NH semi-preparative columns using hexane–diethyl ether–triethylamine (95:5:0.5) as eluent.

TABLE I

k' VALUES OF LEUKOTRIENE A METHYL ESTER ISOMERS

Isomer	*k' values*	
	Spherisorb S5W column	*Spherisorb S5NH column*
2a	3.0	4.3
2b	3.3	4.7
2c	3.5	4.9
2d	3.8	5.3
2e	2.8	4.3
2f	3.0	4.6
2g	3.8	4.7
2h	3.8	4.9
5	4.3	5.9

The k' values of 2a–2h and 5 on the S5W and S5NH columns are shown in Table I. Over 10 mg of isomers 2a–2c, 5 mg of isomer 2d and approximately 20 μg of the minor isomers 2e–2h were isolated. The purity of the isomers isolated was >99% on re-analysis, with the exception of 2c which contained the tetraene (5). All eight isomers were characterised by MS and UV although only the major isomers were analysed by nuclear magnetic resonance (NMR).

Mass spectrometry

Isomers 2a–2h show very similar electron impact mass spectra (Fig. 3 for 2b) with the molecular ion having a significant relative abundance. Although the mass

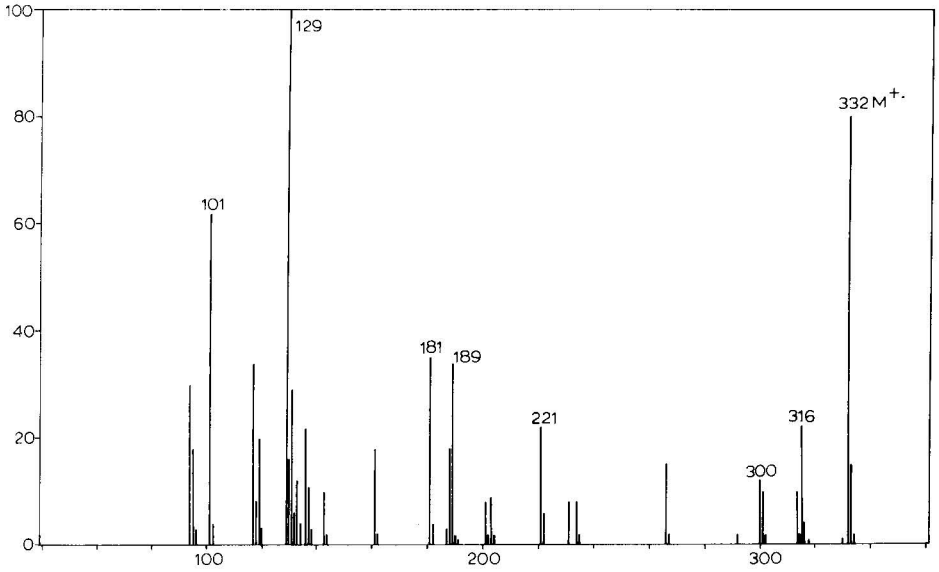
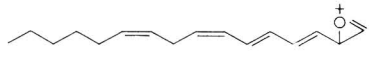
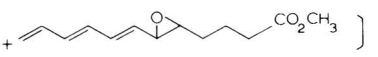
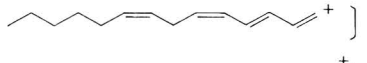
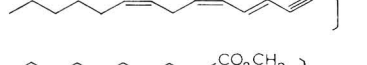
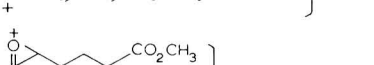
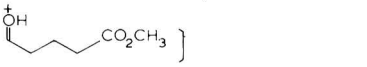
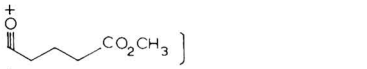
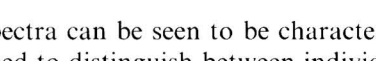


Fig. 3. Electron impact mass spectrum of isomer 2b.

TABLE II

RELATIVE ABUNDANCES OF SIGNIFICANT IONS IN THE MASS SPECTRA OF 2a–2h AND 5

Ion m/e value	*Ion assignment for isomers 2a–2h*	*Isomers*								
		2a	*2b*	*2c*	*2d*	*2e*	*2f*	*2g*	*2h*	*5*
332	$M^{+\cdot}$	80	80	86	100	83	81	86	42	100
316	$[M\text{-}O]^+$	3	22	32	4	3	3	3	2	1
314	$[M\text{-}H_2O]^{+\cdot}$	6	10	2	8	23	1	2	1	1
301	$[M\text{-}OCH_3]^+$	8	10	7	15	8	6	8	3	4
300	$[M\text{-}HOCH_3]^{+\cdot}$	7	12	6	5	8	3	3	4	1
231		6	8	8	5	3	3	5	1	11
221		33	22	14	21	3	10	9	1	3
189		37	34	30	20	9	20	12	3	36
188		25	18	20	8	9	12	5	2	10
181		44	35	22	33	3	16	10	1	1
143		13	10	26	2	5	8	5	11	70
131		30	29	44	10	12	24	10	4	56
129		100	100	100	95	100	100	100	100	41
117		35	34	48	8	11	35	11	4	78
101		60	62	63	50	47	79	47	24	17

spectra can be seen to be characteristic of the molecules concerned, they cannot be used to distinguish between individual isomers. Table II indicates that all molecules show a common elimination of [O], $[H_2O]$, $[OCH_3]$ and $[CH_3OH]$ from the molecular ion. The base ion (*m/e* 129) probably has an acylium ion structure, whereas the *m/e* 131 ion arises from a transannular cleavage[9] with hydrogen transfer. α-Cleavage[10] leads to the formation of intense and diagnostic ions of *m/e* 101 and 231 or *m/e* 143 and 189.

The mass spectrum of the conjugated tetraene 5 differs from those of the other eight isomers in that the *m/e* 131 ion is more favourably formed than the *m/e* 129 ion. α-Cleavage of the 5–6 carbon bond occurs in preference to that of the 4–5 carbon bond, as shown by the increased relative abundance of the *m/e* 143 ion over the *m/e* 101 ion.

UV spectroscopy

The UV absorption maxima in cyclohexane of the eight isomers 2a–2h and 5

TABLE III

UV ABSORPTION MAXIMA OF LEUKOTRIENE A METHYL ESTER ISOMERS

Isomer	$\lambda_{max.}$ *(nm)*			
2a	268.0(sh)	277.5	287.0(sh)	
2b	271.0(sh)	280.5	292.5	
2c	271.5(sh)	280.0	291.5	
2d	267.0	277.5	289.5	
2e	269.0	277.5	288.5	
2f	270.5	281.5	293	
2g	271.0	281.0	292.5	
2h	270.0	281.5	293.5	
5	281.0(sh)	292.0	305.0	320

are listed in Table III. Isomers 2a–2h exhibited a characteristic triene chromophore and 5 was characteristic of a tetraene. The similarity of certain isomers suggests that they are isomeric about the C-14 double bond although it is difficult to draw definite conclusions.

TABLE IV

STEREOCHEMISTRY FOR ISOMERS 2a–2d

Isomer	*Epoxide*	*Double bonds*			
	5,6	*7–8*	*9–10*	*11–12*	*14–15*
2a	*trans*	*trans*	*cis*	*trans*	*cis*
2b	*trans*	*trans*	*trans*	*cis*	*cis*
2c	*trans*	*trans*	*cis*	*cis*	*cis*
2d	*trans*	*trans*	*trans*	*trans*	*cis*

Stereochemistry

After initial MS and UV characterisation of 2a–2d larger samples were collected for NMR analysis. The detailed ^{1}H NMR spectra assignment for 2a–2c determined at 270 MHz have been reported previously[7]. The isomer 2d was later shown by 360 MHz ^{1}H NMR to be the 7,9,11-*trans*-14-*cis*-isomer. The full double bond stereochemistry for 2a–2d is shown in Table IV.

It is envisaged that the use of semi-preparative HPLC will have wide applications in the purification of further members of the leukotriene family. Further transformations of compounds 2a–2d to isomers of leukotrienes B, C, D and E and measurement of their biological activities will be the subject of future communications.

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Note

Preparative ion-pair high-performance liquid chromatography and gas chromatography of pyrrolizidine alkaloids from comfrey

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Interest in methods for the analysis of pyrrolizidine ester alkaloids (PAs) has increased considerably since it was discovered that many of them possess hepatotoxic properties[1] and that some of them may have potential use as cytostatic agents in cancer therapy[2].

For this analysis, namely thin-layer chromatography (TLC)[1–5], column chromatography[6–10] and counter-current distribution[9] have been used, and to a lesser extent paper electrophoresis[9,11] and gas chromatography[12–16]. However, detailed studies of the gas chromatographic separation of mono- and diester alkaloids in complex mixtures have not so far been published. In our laboratory, a rapid derivatization procedure for PAs prior to analytical and capillary gas chromatographic (GC) separation was developed. The PA derivatives gave mass spectra that were valuable in the qualitative evaluation of the separated PAs.

A search for faster column chromatographic separations of PAs led to the development of high-performance liquid chromatographic (HPLC) methods[17–20]. An extension of these methods to preparative HPLC has so far only been utilized for the separation of macrocyclic PAs from *Senecio vulgaris*[21], but the method was laborious and expensive.

In this paper we describe a cheap and convenient method for the preparative ion-pair HPLC separation of mono- and diester PAs from comfrey, based on the ion-pair separation described previously for TLC[5].

EXPERIMENTAL

Pyrrolizidine alkaloids from ground comfrey roots were isolated as described previously[22]. All reagents and solvents were of analytical-reagent grade.

For preparative ion-pair HPLC a 53 × 2.5 cm I.D. stainless-steel column (Waters Assoc., Milford, MA, U.S.A.) was packed with dry silica gel 60 (230–400 mesh) from Merck (Darmstadt, G.F.R.) under continuous vibration with an engraving instrument. After connection of this column to a Waters Preparative LC/System 500 A, flushing with an eluent consisting of 0.075 *M* lithium chloride in chloroform–methanol (85:15) was carried out until the refractive index (RI) remained constant. Then the column was loaded with a solution of 750 mg of root extract in 10 ml of the eluent and the alkaloids were separated at a flow-rate of 50 ml/min.

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Fractions that coincided with monitored RI peak profiles were collected and the eluent was removed *in vacuo* at 50°C by means of a rotary evaporator. The alkaloids were dissolved in chloroform, leaving the remaining lithium chloride behind.

Approximately 1 mg of the HPLC-purified alkaloid (after evaporation of chloroform in a stream of air) was dissolved in 30 μl of chloroform–dimethyl sulphoxide (1:1) and derivatized with 75 μl of a freshly prepared mixture of hexamethyldisilazane and trimethylchlorosilane (10:1) at room temperature for 5 min in a PTFE-lined Sovirel tube. Whenever small amounts of water were still present in the samples prior to derivatization, 1 ml of dimethoxypropane was added as a water scavenger and removed again by passing a stream of nitrogen into the derivatization tube.

Separation of the trimethylsilyl (TMS) ethers was achieved in a Packard 429 gas chromatograph equipped with a flame-ionization detector on a 1400 × 0.4 cm I.D. glass column packed with 4% OV-17 on Chromosorb W HP (100–120 mesh) under the following conditions: nitrogen flow-rate, 25 ml/min; detector block temperature, 300°C; injector temperature, 250°C; initial column temperature, 220°C, increased at 3.5°C/min to a final temperature of 270°C.

Combined GC–mass spectrometry (MS) was performed on a Finnigan 3300 quadrupole mass spectrometer equipped with a standard chemical ionization source and a 6110 data system at an electron energy of 70 eV and an ionizing current of 100 μA. In this instance a wide-bore 2500 × 0.05 cm I.D. CpSil 5 column (Chrompack, Middelburg, The Netherlands) was used under the following conditions: initial column temperature, 200°C, increased at 4°C/min to a final temperature of 330°C. Mass spectra were taken with a 1.87-sec cycle time and data acquisition was started after 1.85 min.

RESULTS AND DISCUSSION

Fig. 1 illustrates the preparative ion-pair HPLC separation of the pyrrolizidine alkaloids of comfrey roots. The elution pattern resembled the pattern found previously for ion-pair TLC[5]. Lycopsamine/intermedine (peak A) was eluted first, followed by acetyllycopsamine/acetylintermedine (peak B) and symphytine isomers (peak C), as confirmed by comparison with reference substances. No recycling was needed for adequate separation, resulting in short analysis times.

After evaporation of the eluent *in vacuo* the alkaloids were easily separated from the remaining lithium chloride by dissolving them in chloroform. The combined evaporated organic phases were purified by distillation using a Hempel column and checked for purity and deviation from the original 85:15 of the chloroform to methanol ratio by gas chromatography. By addition of more of one of these solvents the correct 85:15 ratio was obtained again. This recycling of solvents and the low cost of the silica gel columns made the entire method cheap in comparison with reversed-phase separations. Another feature of the described system over reversed-phase systems is that alkaloids are simply isolated by evaporation of solvent only, so no basification and extraction of eluted fractions from water–methanol mixtures, which can cause hydrolysis of ester alkaloids, is needed.

The alkaloids from the collected fractions A, B and C were derivatized and separated by gas chromatography on a packed OV-17 column. The short derivatization time at room temperature with the chosen mild silylation agent did not give a

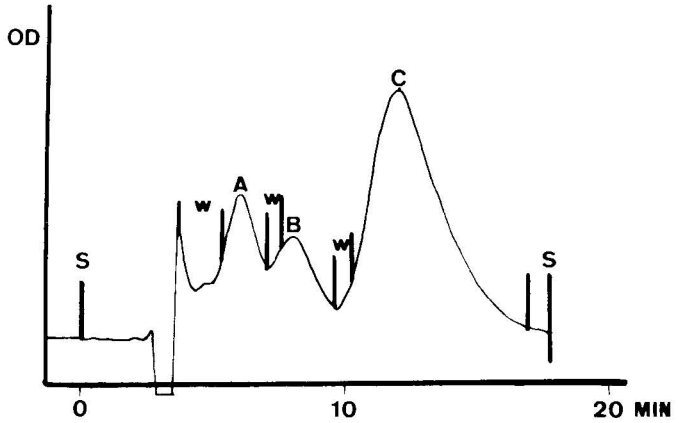


Fig. 1. Preparative HPLC of pyrrolizidine alkaloids from comfrey as ion pairs. Column, 53 × 2.5 cm I.D., packed with silica gel 60 (230–400 mesh); eluent, 0.075 *M* lithium chloride in chloroform–methanol (85:15); flow-rate, 50 ml/min. OD = optical density; S = injection (750 mg of alkaloid in 10 ml of the eluent); W = waste; A = lycopsamine + intermedine; B = acetyllycopsamine + acetylintermedine; C = symphytine and/or isomers.

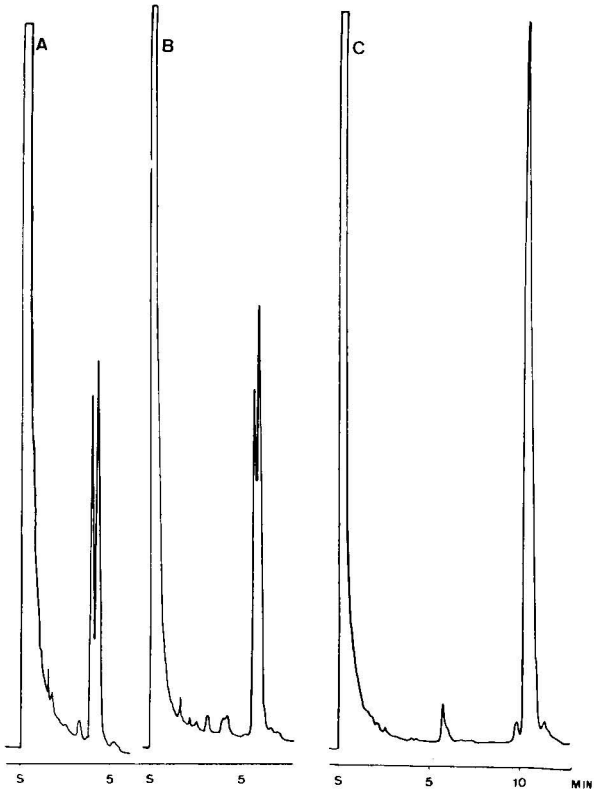


Fig. 2. GC analysis of TMS ethers of the pyrrolizidine alkaloid fractions A–C that eluted from the preparative HPLC as ion pairs (compare with Fig. 1).

complete reaction with the hydroxyl groups of the esterifying acids, as will be shown later. However, this did not affect the separation patterns when the derivatization time did not exceed 30 min. A longer derivatization period gave rise to the appearance of additional peaks, probably because of a higher degree of derivatization of the alkaloids.

Fig. 2 shows the GC separation of the alkaloids in the various HPLC fractions. The double peaks are due to the presence of stereoisomeric forms of the alkaloids. The separation of these isomers by means of affinity chromatography will be described elsewhere.

A total separation of lycopsamine/intermedine and acetyllycopsamine/acetylintermedine together with a partial separation of the isomers of symphytine could be shown by capillary GC on CpSil 5 columns (Fig. 3) in combination with MS. The mass spectra of the isomers were similar in their fragmentation, as would be expected, although the relative intensities of fragments could differ (Fig. 4). The fragmentation pattern of lycopsamine/intermedine (L/I) was comparable to that of indicine N-oxide in the pyrrolizidine form (INOPYZ) except for the ion at *m/e* 261, which was absent in the L/I mass spectra[16]. This peak should be indicative of the fully silylated esterifying dihydroxy acid. The peak at *m/e* 117 (relative intensity 51.5%) shows, however, that the secondary hydroxyl group was silylated (compare Table I).

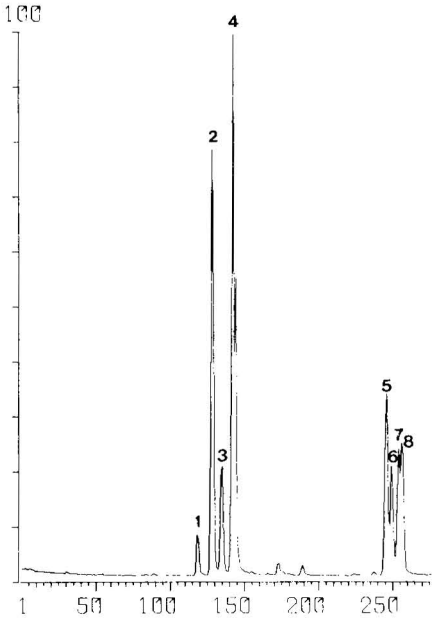


Fig. 3. Wide-bore capillary GC of TMS ethers of pyrrolizidine alkaloids from comfrey root on CpSil 5. 1, 2 = Lycopsamine/intermedine; 3, 4 = acetyllycopsamine/acetylintermedine; 5, 6, 7, 8 = symphytine and isomers.

This, albeit incomplete, silylation was enough to ensure sufficient volatility. Peaks in the mass spectra of the distinct TMS-alkaloids at *m/e* 210, 180 and 220, gave full information of the substitution level at C-7: TMS, acetyl, or tiglic/angelic acid respectively, for L/I, acetyl-L/I and symphytine isomers (Figs. 4 and 5).

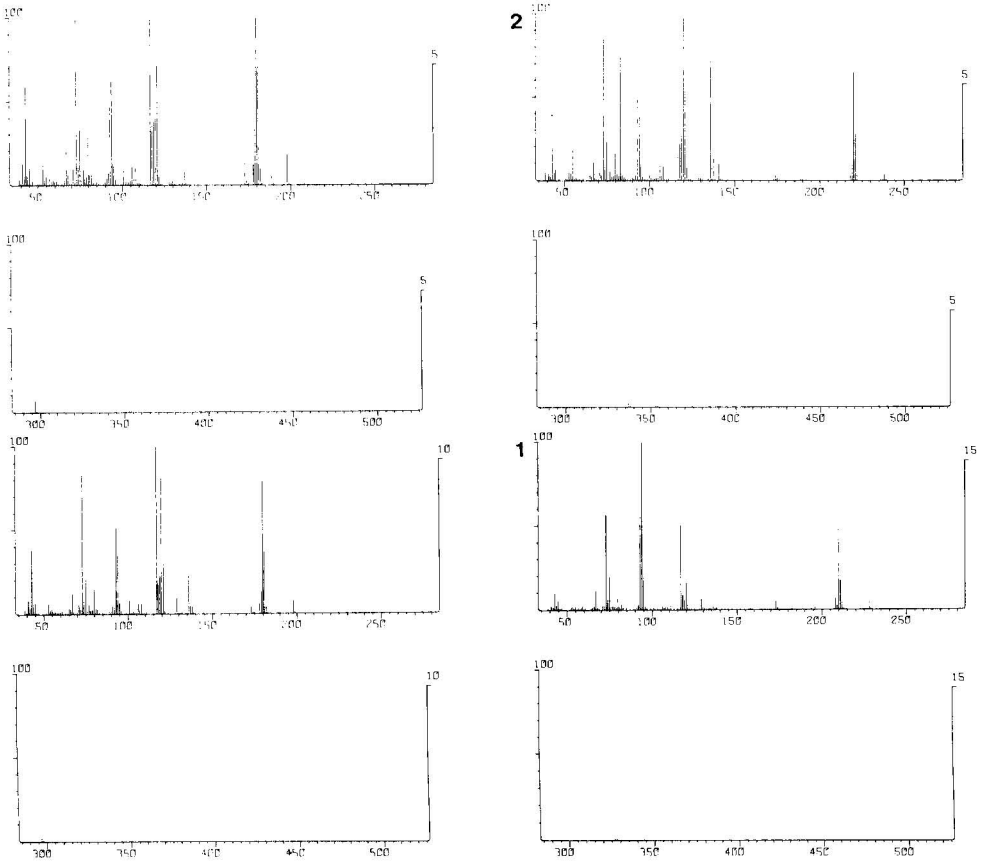
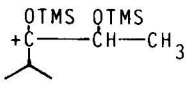
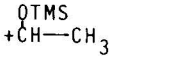


Fig. 4. Mass spectra of diastereoisomeric TMS-acetyllycopsamine or TMS-acetylintermedine (GC peaks 3 and 4, respectively, in Fig. 3).

Fig. 5. Mass spectra of TMS_2-lycopsamine (1) or diastereoisomer and TMS-symphytine (2) or isomer. Compare also with Fig. 4.

TABLE I

FRAGMENTS IN THE MASS SPECTRA OF INDICINE N-OXIDE IN THE PYRROLIZIDINE FORM (INOPYZ) AND LYCOPSAMINE/INTERMEDINE (L/I) TRIMETHYLSILYL DERIVATIVES, SHOWING THE DIFFERENCE IN DERIVATIZATION SITE

Fragment	*m/e*	*Relative abundance (%)*	
		INOPYZ	L/*I*
	261	7.64	–
	117	30.10	51.5

We believe that these methods can contribute to the faster and easier analysis of pyrrolizidine alkaloids from different plant sources, which is of importance for pharmacognostic screenings of plants that show hepatotoxic properties and for chemosystematics.

ACKNOWLEDGEMENTS

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Note

Separation of amines, guanidines and hydroxycinnamic acid amides by ion-exchange chromatography

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For the continuation of a study on amine metabolism in barley seedlings, it was necessary to develop a method for separating *p*-coumarylagmatine and its antifungal dimers (the hordatines A and B) and their glycosides (hordatines M) (Fig. 1)[1,2]. Di- and polyamines can be separated by the widely used procedure of automated ion-exchange chromatography[3] with detection by fluorescence after reaction with *o*-phthalaldehyde[4]. Although coumarylagmatine and the hordatines cannot be detected by the *o*-phthalaldehyde system, the guanidino groups in these compounds will form an orange chromogen with the Sakaguchi reagent. An amine analyser was therefore designed with the column eluate divided between two systems, using respectively *o*-phthalaldehyde derivatisation for amines and the automated Sakaguchi reaction for guanidines. Similar automated Sakaguchi systems have been described in the separation of more acidic unconjugated guanidino compounds[5,6]. Elution of the hordatines from a cation-exchange resin requires the use of buffers of higher pH and ionic strength than are necessary for the separation of the polyamines. This may be due to hydrogen bonding of the aromatic rings to the polystyrene resin.

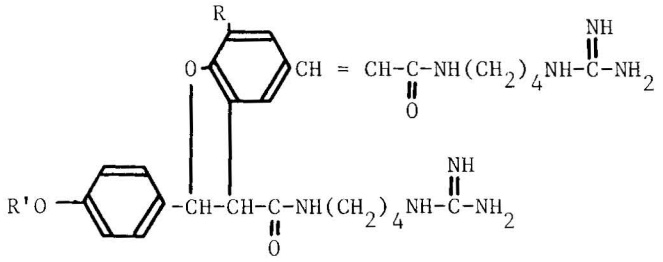

Fig. 1. Structures of the hordatines, antifungal dimers of coumarylagmatine found in barley seedlings.
1 Hordatine A: R = H, R′ = H
2 Hordatine B: R = OCH_3, R′ = H
3 Hordatines M: R = OCH_3 or H, R′ = D-glucopyranosyl.

EXPERIMENTAL

Sample preparation

For preparation of extracts, barley shoots were soaked for 18 h in 4 volumes 18

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TABLE I

BUFFER COMPOSITION AND ELUTION CYCLE

Buffer	*pH*	*Composition*	*Temperature (°C)*	*Duration (min)*	*Compounds eluted*
1	3.0	0.2 *M* Na_3 citrate 0.1 *M* NaCl	56	24	Acidic and neutral amino acids
2	5.6	0.2 *M* Na_3 citrate 0.1 *M* NaCl	56	30	Basic amino acids
3	5.6	0.2 *M* Na_3 citrate 2.0 *M* NaCl	75	39	Polyamines
4	10	0.2 *M* $NaHCO_3$ 2.5 *M* NaCl 0.08 *M* NaOH	75	27	Coumarylagmatine
5	13	0.05 *M* NaCl 0.13 *M* NaOH	95	30	Hordatines A and B
6	13	0.45 *M* NaCl 1.17 *M* NaOH	95	30	Hordatines M

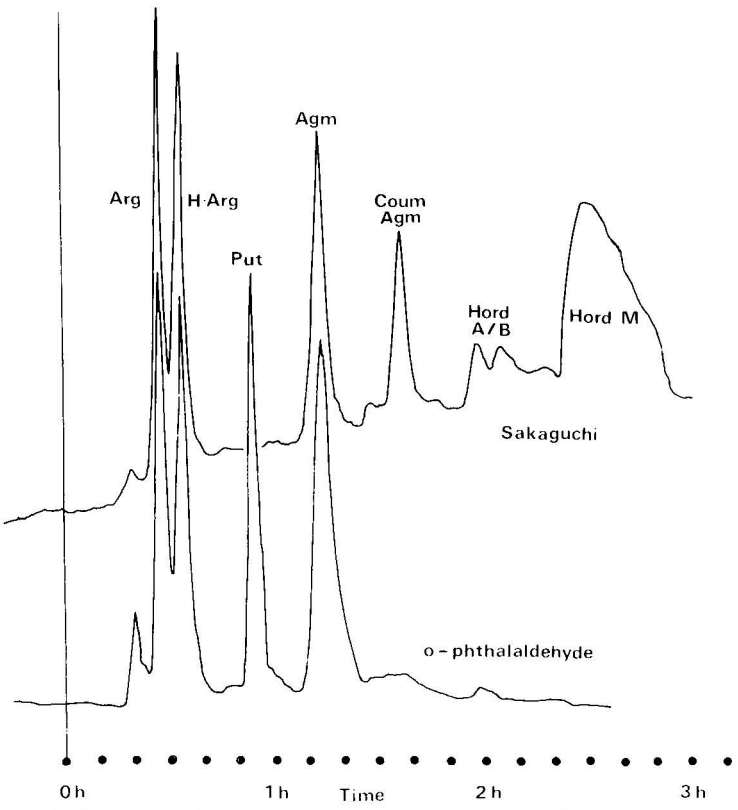

Fig. 2. Separation of amino acids, amines and guanidines by the amine analyser, with detection by *o*-phthalaldehyde and Sakaguchi reagents. Arginine (Arg), L-homoarginine (H·Arg), putrescine (Put) and agmatine (Agm) were authentic standards. Coumarylagmatine (Coum.Agm) and the hordatines (Hord) were extracted from the shoots of barley seedlings.

M acetic acid at 4°C. The filtrate was evaporated to dryness at less than 40°C, dissolved in 0.1 volume water and clarified by centrifugation.

Chromatographic procedure

A sequence of 6 buffers was developed for optimal resolution of the relevant compounds (Table I). All buffers contained Hibitane (10 mg/l) to prevent microbial growth and were passed through a 0.22-μm Millipore filter. Buffer 1 was pumped for 15 min to equilibrate the column prior to sample injection. A maximum sample volume of 0.25 ml was injected, containing 0.1 μmole L-homoarginine as an internal standard. This is eluted immediately following arginine (Fig. 2). Buffers were selected by a six-port automated rotary valve and passed through the column at 0.5 ml/min by a Milton Roy minipump. The back-pressure fell from 4 to 1.4 MPa during the cycle. The 13 × 0.4 cm stainless-steel column was packed with 8–9 μm Durrum DC4A sulphonated polystyrene cation-exchange resin. Column temperature was regulated in 3 steps at 56°C, 75°C and 95°C by immersion in a programmed water-bath. The

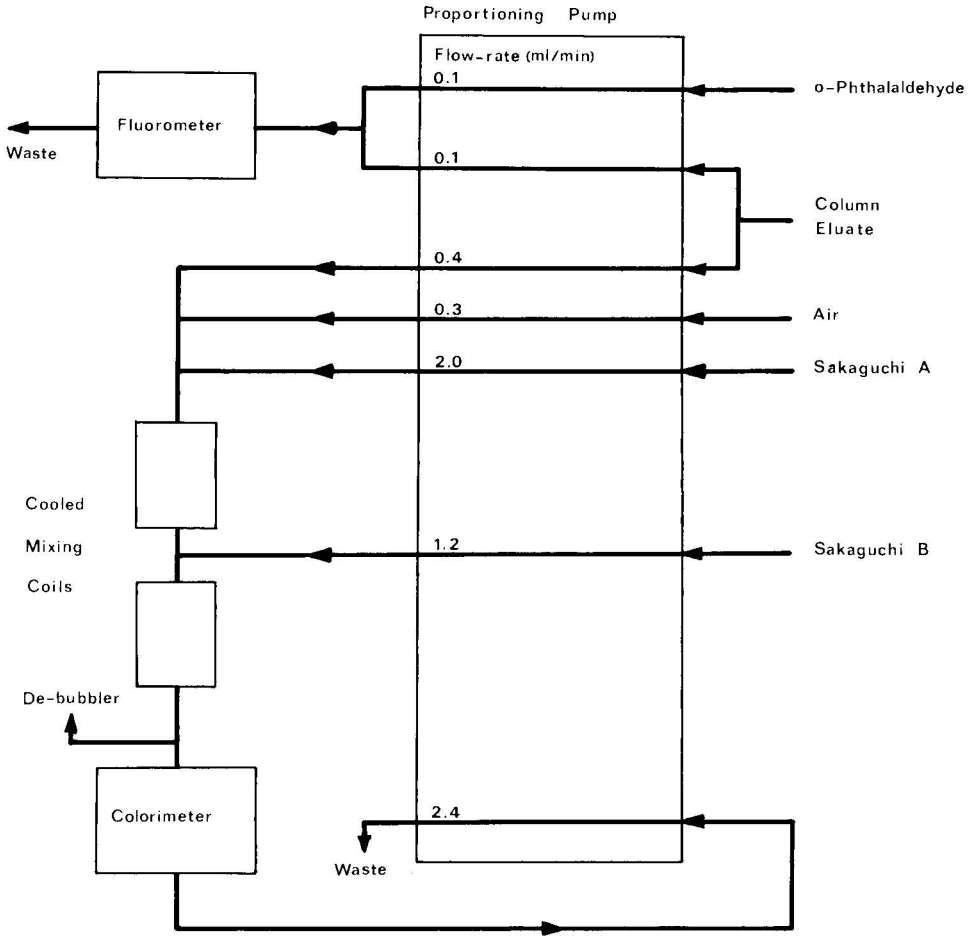

Fig. 3. Post-column flow diagram for the amine analyser.

elution cycle was controlled by a programmer which operated the buffer supply valve and regulated the column temperature.

Detection systems

The column eluate was divided between the two detection channels and the reagents added by a proportioning pump (Fig. 3). *o*-Phthalaldehyde reagent[4] was mixed at an equal flow-rate (0.1 ml/min) with 20% of the eluate, and the fluorescence monitored after a 2-sec delay in a Gilson Spectra/glo fluorometer equipped with a 40-μl flow cell (excitation 360 nm, emission 455 nm). *o*-Phthalaldehyde reagent was stored at 4°C under nitrogen. The remainder of the column eluate (0.4 ml/min) was segmented with air bubbles (0.3 ml/min) and mixed with 8-hydroxyquinoline sulphate (3.4 m*M* in 3 *M* NaOH, 2 ml/min, Sakaguchi A) in a coil at 4°C. N-Bromosuccinimide (8.4 m*M*, 1.2 ml/min, Sakaguchi B) was then added and mixing was completed in a further coil at 4°C. The air bubbles were removed and the flow fed to a colorimeter for monitoring at 495 nm.

CONCLUSIONS

The response of the fluorescence channel is linear between 10 pmole and 1 μmole of standard amine injected. However in the present work this detection system was considerably attenuated since the Sakaguchi channel is of much lower sensitivity and is limited to a linear range of 10 nmole to 0.25 μmole.

Separation of the compounds related to hordatine metabolism could be achieved in *ca.* 3 h with little sample preparation (Fig. 3).

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Note

Purification of potassium phosphate for high-performance liquid chromatography

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Potassium phosphate buffers, while having several advantages over other solvent systems for many high-performance liquid chromatography (HPLC) applications, suffer from a serious disadvantage, namely a high background absorption in the UV region. This is true for reagent-grade phosphates of every supplier tested. The problem becomes particularly acute when very small amounts of substances requiring high concentrations of buffer for elution are to be separated, a typical example being the separation of nucleoside triphosphates on ion-exchange columns. Although this absorption is obviously due to an impurity, several methods of purification fail to correct the problem. The only published method[1] for this purpose involves a Dowex 1-X8 purification and a series of time- and material-consuming recrystallizations.

We have been successful in cleaning up our phosphate buffers by the use of Chelex-100, a Bio-Rad (Richmond, CA, U.S.A.) chelating carboxylic acid cation exchanger. Chelex-100 is a styrene–divinylbenzene copolymer containing paired iminodiacetate ions which chelates preferentially transition metals, even in highly concentrated salt solutions. We are routinely passing an 1 *M* solution of KH_2PO_4 through a column of Chelex-100 (100–200 or 200–400 mesh) at a rate of about 1 ml/min · cm^2 of bed. This treatment effectively removes most of the UV absorbance from the phosphate, to the extent that a gradient reaching 1 *M* concentration can be used with full scale absorbance at 254 nm of 0.08 or 0.04 with a baseline shift of less than 5% or 10%, respectively.

The Chelex-100 is suspended directly in 1 *M* KH_2PO_4 and the slurry used to pack the column. The eluent is collected after 100 ml have passed through. Over 20 liters of 1 *M* solution can be purified through a 120-ml (250 × 25 mm) column without regeneration of the resin. Thus, the process is very economical even if the resin is discarded after use, but regeneration is possible following the manufacturer's instructions.

Fig. 1 represents a comparison of the UV spectra of the 1 *M* KH_2PO_4 solution before and after Chelex-100 purification. The nature of the material removed by the treatment has not been investigated. It is, however, of interest to note that the impurity accumulates on anion-exchange columns at concentrations lower than 1 *M*. Thus, if several runs are performed with gradients reaching up to 0.60 or 0.70 *M*, a subsequent 1 *M* elution will slowly remove large amounts of UV absorbing material. For the same reason, even if a concentration of 1 *M* is not routinely reached

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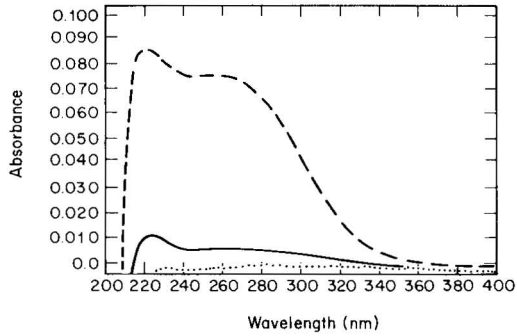

Fig. 1. UV spectra of 1 *M* KH_2PO_4 before and after purification with Chelex-100. Both unpurified (broken line) and purified (solid line) solutions were read against distilled water which had been passed through a Chelex-100 column. Distilled water not subjected to Chelex-100 treatment gave the spectrum indicated by the dotted line.

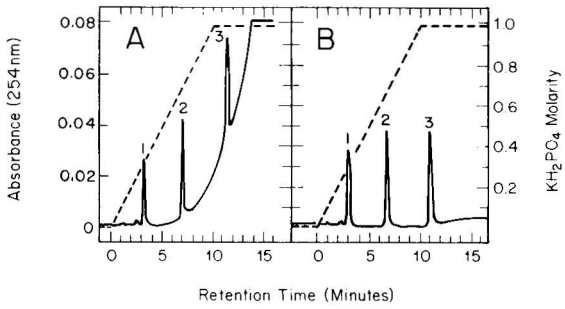

Fig. 2. Comparison of baseline quality before and after purification of KH_2PO_4 by Chelex-100. Instrument: Varian 5020; column: Varian Micropak AX-10, 30 cm × 4 mm I.D.; flow-rate, 3 ml/min; program: from 10 m*M* to 1 *M* KH_2PO_4 in 10 min. Peaks: 1 = AMP (0.3 nmoles); 2 = ADP (0.4 nmoles); 3 = ATP (0.6 nmoles). Full scale absorbance, 0.08. A, unpurified KH_2PO_4; B, purified KH_2PO_4.

in a particular separation, the quality of the baseline progressively deteriorates when unpurified buffers are used. A prolonged purge with purified buffer will eventually restore a stable low baseline.

The dramatic effect of Chelex-100 purification on baseline quality is illustrated in Fig. 2, which compares the separation of nanomole quantities of AMP, ADP and ATP on an AX-10 (Varian, Palo Alto, CA, U.S.A.) anion-exchange column with a 0.01 to 1 *M* gradient of KH_2PO_4, before and after treatment. The baseline goes off-scale with the unpurified buffer (Fig. 2A), while it increases by only 4% with the Chelex-purified buffer (Fig. 2B). Thus, picomole amounts of triphosphates can be accurately determined, without elaborate and uncertain baseline corrections, using Chelex-purified buffers.

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Gas Chromatography

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2. FUNDAMENTALS, THEORY AND GENERAL

2b. Thermodynamics and theoretical relationships

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See 3969, 3971, 4017, 4037, 4095.

32e. Plant extracts

See 4041.

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See 3912.

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See also 4091.

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See 4083, 4084.

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19. PROTEINS

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See also 4427.

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21. PURINES, PYRIMIDINES, NUCLEIC ACIDS AND THEIR CONSTITUENTS

21a. Purines, pyrimidines, nucleosides, nucleotides

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35. SOME TECHNICAL PRODUCTS AND COMPLEX MIXTURES

35a. Surfactants

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20. ENZYMES

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21. PURINES, PYRIMIDINES, NUCLEIC ACIDS AND THEIR CONSTITUENTS

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23.OTHER SUBSTANCES CONTAINING HETEROCYCLIC NITROGEN

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24. ORGANIC SULPHUR COMPOUNDS

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30. SYNTHETIC AND NATURAL DYES

30b. Chloroplast and other natural pigments

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32. PHARMACEUTICAL AND BIOMEDICAL APPLICATIONS

32f. Clinico-chemical applications and profiling body fluids

See 5577, 5583, 5584, 5687.

33. INORGANIC COMPOUNDS

33a. Cations

5756 Gebauer, P., Bocek, P., Deml, M. and Janak, J.: Isotachophoresis of kinetically labile complexes. *J. Chromatogr.*, 199 (1980) 81-94.

35. SOME TECHNICAL PRODUCTS AND COMPLEX MIXTURES

35a. Surfactants

5757 Despotovic, R. and Tomasic, V.: On surfactant/surfactant systems in aqueous media. *Tensioactivos: Biodegradabilidad, Fis.-Quim. Apl., Jorn. Com. Esp. Deterg., 11th*, (1980) 475-485; *C.A.*, 93 (1980) 156216f.

35c. Various technical products

5758 Donath, E. and Pastusenko, V.: (Electrophoretic properties of particles covered by polymers). *Acta Polym.*, 31 (1980) 537-538; *C.A.*, 93 (1980) 201479g.

35d. Complex mixtures and non-identified compounds

5759 Redgwell, R.J.: Fractionation of plant extracts using ion-exchange Sephadex. *Anal. Biochem.*, 107 (1980) 44-50.

36. CELLS AND CELLULAR PARTICLES

5760 Campbell, K.P., Franzini-Armstrong, C. and Shamoo, A.E.: Further characterization of light and heavy sarcoplasmic reticulum vesicles. Identification of the "sarcoplasmic reticulum feed" associated with heavy sarcoplasmic reticulum vesicles. *Biochim. Biophys. Acta*, 602 (1980) 97-116 - polyacrylamide gel, SDS-polyacrylamide gel.

5761 Catsimpolas, N.: Principles and instrumentation of analytical cell electrophoresis. *Electrophoresis*, 1 (1980) 73-78; *C.A.*, 93 (1980) 217329u.

5762 Colas, B. and Maroux, S.: Simultaneous isolation of brush border and basolateral membrane from rabbit enterocytes. Presence of brush border hydrolases in the basolateral membrane of rabbit enterocytes. *Biochim. Biophys. Acta*, 600 (1980) 406-420 - crossed immunoelectrophoresis.

5763 Fujita, S.: Further studies on the smooth-surfaced membranes isolated from rat brain. *J. Biochem.*, 88 (1980) 1615-1622 - SDS-polyacrylamide gel.

5764 Hackney, D.D.: Photodynamic action of bilirubin on the inner mitochondrial membrane. Implications for the organization of the mitochondrial ATPase. *Biochem. Biophys. Res. Commun.*, 94 (1980) 875-880 - polyacrylamide gel.

5765 Jelenc, P.C.: Rapid purification of highly active ribosomes from *Escherichia coli*. *Anal. Biochem.*, 105 (1980) 369-374.

5766 Lester, E.P., Lemkin, P., Lipkin, L. and Cooper, H.L.: Computer-assisted analysis of two-dimensional electrophoreses of human lymphoid cells. *Clin. Chem.*, 26 (1980) 1392-1402; *C.A.*, 93 (1980) 200353z.

5767 McGuire, J.K., Miller, T.Y., Tipps, R.W., Snyder, R.S. and Righetti, P.G.: New experimental approaches to the isoelectric fractionation of cells. *J. Chromatogr.*, 194 (1980) 323-333.

5768 Rustin, P., Julienne, M. and Kader, J.C.: (Free-flow electrophoresis isolation of mitochondria from spinach leaves (*Spinacia oleracea* L.)). *C.R. Acad. Sci., Ser. D*, 291 (1980) 105-108; *C.A.*, 93 (1980) 217389p.

5769 Takano, S., Itagaki, S., Sakurai, K. and Suzuki, T.: Influences of prostaglandins on electrophoretic mobility and aggregation of rabbit platelets. *Prostaglandins*, 20 (1980) 579-586; *C.A.*, 93 (1980) 198175x.

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journal of
chromatography news section

NEW BOOKS

Data processing in chemistry (A collection of papers presented at the Summer School, Rzeszo'w, Poland, August 24–31, 1980), edited by Z. Hippe, Elsevier, Amsterdam, Oxford, New York, 1981, X + 288 pp., Dfl. 150.00, US$ 73.25, ISBN 0-444-99744-X.

Detection and measurement of hazardous gases, edited by C.F. Cullis and J.G. Firth, Heinemann, London, Exeter, NH, 1981, X + 226 pp., price £ 25.00, ISBN 0-435-71030-3.

Advances in hemoglobin analysis, edited by S.M. Hanash and G.J. Brewer, Heyden, London, 1981, 211 pp., price £ 15.00, DM 66.00, ISBN 0-8451-0060-2.

MEETINGS

INTERNATIONAL SYMPOSIUM ON CHROMATOGRAPHY AND MASS SPECTROMETRY IN BIOMEDICAL SCIENCES

The Italian Group for Mass Spectrometry in Biochemistry and Medicine, in co-operation with the International Scientific Center, is organizing an "International Symposium on Chromatography and Mass Spectrometry in Biomedical Sciences" on June 21–23, 1982, in Bordighera, Italy.

The symposium will discuss all the latest aspects of chromatography, mass spectrometry and chromatography–mass spectrometry and their areas of applications, including biochemistry, medicine, toxicology, drug research, forensic science, clinical chemistry and pollution. The symposium will consist of presentations from eminent invited speakers and free communications. Facilities will be available for participants to display poster communications.

An instrument and publication exhibition will be held throughout the Symposium.

For further details, please contact: Dr. Alberto Frigerio, c/o Istituto di Ricerche Farmacologiche "Mario Negri", Via Eritrea, 62 – 20157 Milan, Italy. Tel.: 35.54.546; Telex: 331268 NEGRI I.

SAC '83, INTERNATIONAL CONFERENCE AND EXHIBITION ON ANALYTICAL CHEMISTRY

SAC '83, organised by the Analytical Division of the Royal Society of Chemistry, will be held from July 17th to the 23rd, 1983, at the University of Edinburgh in Scotland. The scientific programme will be organised around plenary, invited and contributed papers and posters covering the whole field of Analytical Chemistry. In addition, there will be 1-day Symposia on particular Analytical themes organised by RSC Groups and other associated bodies. The programme will include a series of Workshops, where research workers can demonstrate new apparatus and techniques, as well as informal evening discussion meetings and one-day UPDATE courses.

Delegates may register for the whole conference or, if they are members of the Royal Society of Chemistry, for individual days. Registration for the UPDATES is separate. Students, accompanying and retired persons may attend the whole conference at a reduced fee.

(Continued on next page)

An extensive exhibition of commercial apparatus, equipment and books will be a major feature of the conference. Most of the exhibits will be immediately adjacent to the Lecture Theatres and poster area, and lecture-free times will be organised to facilitate attendance at the exhibition.

For further information contact The Secretary, Analytical Division, Royal Society of Chemistry, Burlington House, London W1V 0BN, Great Britain.

SHORT COURSES

BASIC PROGRAMMING COURSES FOR CHROMATOGRAPHERS

Laboratory Data Control announces new one-day intensive courses in BASIC programming and its applications in chromatography, both GC and HPLC. Given by the LDC Automation Products staff, the course is designed for both experienced programmers and chromatographers getting their first exposure to data handling by BASIC. The novice will learn the fundamentals of BASIC and its application to chromatography while the experienced programmer will gain "hands-on" exposure to state-of-the-art instrumentation.

Key topics discussed include: an introduction to BASIC; example chromatographic applications; using programs from a "program library"; essential concepts of programming such as: data storage and retrieval, data manipulation and calculation, display and output of results; programming examples; and, applying BASIC to future applications.

Tuition is US$50.00 and includes textbook, manual, example programs and lunch.

Courses are currently scheduled for California, Texas, Oklahama, New Jersey, Alabama and Tennessee. For further information on these or future courses in other locations contact: Lois Lucciola, telephone: (800) 327-6182 (from inside the U.S. only); or Laboratory Data Control, Division of Milton Roy Co., P.O. Box 10235, Riviera Beach, FL 33404, U.S.A. Telephone (305) 844-5241; TELEX 513479.

CALENDAR OF FORTHCOMING MEETINGS

Oct. 22–23, 1981 Montreux, Switzerland	**Workshop on Liquid Chromatography – Mass Spectrometry** Contact: Prof. Dr. R.W. Frei, Free University, Department of Analytical Chemistry, De Boelelaan 1083, 1081 HV Amsterdam, The Netherlands (Further details published in Vol. 207, No. 3).
Oct. 27–29, 1981 London, Great Britain	**Petroanalysis 81** Contact: Miss I.A. McCann, Conference Officer, Institute of Petroleum, 61 New Cavendish Street, London W1M 8AR, Great Britain. (Tel: 01–636 1004, Telex: 264380)
Oct. 28–30, 1981 Gatlinburg, TN, U.S.A.	**Resource Recovery and Environmental Issues of Industrial Solid Wastes** Contact: J.S. Watson, Oak Ridge National Laboratory, P.O. Box X, Oak Ridge, TN 37830, U.S.A.
Nov. 9–10, 1981 Berlin, G.F.R.	**Symposium on Practical Aspects of HPLC** Contact: Dr. I. Molnár, Wissenschaftliche Geratebau Dr. H. Knauer GmbH, Hegauer Weg 38, D-1000 Berlin 37, G.F.R. (Further details published in Vol. 207, No. 2).
Nov. 16–17, 1981 Washington, DC, U.S.A.	**International Symposium on HPLC of Proteins and Peptides** Contact: Ms. S.E. Schlessinger, Symposium Manager, 400 East Randolph, Chicago, IL 60601, U.S.A. (Further details published in Vol. 208, No. 2)
Nov. 23–25, 1981 Barcelona, Spain	**2nd International Congress on Analytical Techniques in Environmental Chemistry** Contact: Dr. Joan Albaigés, General Secretary, Plaza de Espana, Barcelona-4, Spain. Tel. 223 31 01.

Nov. 23–28, 1981 Barcelona, Spain	**EXPOQUIMIA 81 (Salón Internacional de la Química)** Contact: EXPOQUIMICA, Plaza de Ẽspana, Barcelona-4, Spain. Tel.: 223 31 01; Telex: 53117 FOIMB-E.
Nov. 25–27, 1981 Barcelona, Spain	**2nd Mediterranean Congress of Chemical Engineering** Contact: 2nd Mediterranean Congress of Chemical Engineering, EXPOQUIMIA-81, Plaza de Ẽspana, Barcelona, Spain. Tel.: (93) 223 31 01; Telex: 53117 FOIMB-E.
Nov. 26–27, 1981 Barcelona, Spain	**Workshop on the Chemistry and Analysis of Hydrocarbons in the Environment** Contact: Workshop office: Dr. J. Albaigés, Expoquimia, Plaza de Ẽspana, Barcelona-4, Spain. Tel.: 223 31 01, ext. 406; Telex: 53117 FOMB-E.
Dec. 2–3, 1981 Paris, France	**Journées de Chromatographie en Phase Liquide** Contact: H. Colin, Laboratoire C.A.P., Ecole Polytechnique, Route de Saclay, 91128 Palaiseau Cedex, France.
Dec. 3rd, 1981 London, Great Britain	**Capillary Columns** Contact: The Royal Society of Chemistry, Analytical Division (Chromatography and Electrophoresis Group), Burlington House, London W1V 0BN, Great Britain.
Dec. 9 + 10, 1981 London, Great Britain	**New Developments and Automated Systems for Sampling and Monitoring Air and Water** Contact: The Royal Society of Chemistry, Analytical Division (Special Techniques Group), Burlington House, London W1V 0BN, Great Britain.
Jan. 4–9, 1982 Orlando, FL, U.S.A.	**1982 Winter Conference on Plasma Spectrochemistry** Contact: 1982 Winter Conference, c/o ICP Information Newsletter, Chemistry -GRC Towers, University of Massachusetts, Amherst, MA 01003, U.S.A. Tel. (413) 545–2294.
Jan. 19–20, 1982 Amsterdam, The Netherlands	**Symposium on "Detection in High-Performance Liquid Chromatography"** Contact: Mrs. Peschier, Hewlett-Packard Nederland B.V., Analytical Department, van Heuven Goedhartlaan 121, 1181 KK Amstelveen, The Netherlands. (Tel.: 020-47 20 21). (Further details published in Vol. 212, No. 2)
Feb. 10th, 1982 Birmingham, Great Britain	**Pattern Recognition in the Analytical Sciences** Contact: The Royal Society of Chemistry, Analytical Division, Burlington House, London W1V 0BN, Great Britain.
Feb. 16 + 17, 1982 Edinburgh, Scotland	**Advances in Chromatography: Industrial and Petrochemical Applications** Contact: The Royal Society of Chemistry, Analytical Division (Scottish Region), Burlington House, London W1V 0BN, Great Britain.
Feb. 18th, 1982 Bristol, Great Britain	**Automated Combination Techniques with Mass Spectrometry** Contact: The Royal Society of Chemistry, Analytical Division (Automatic Methods Group), Burlington House, London W1V 0BN, Great Britain.
Feb. 25th, 1982 Aberdeen, Scotland	**Quantitative Analysis of Environmental Samples by Gas Chromatography–Mass Spectrometry** Contact: The Royal Society of Chemistry, Analytical Division (Scottish Region), Burlington House, London W1V 0BN, Great Britian.

March 8–12, 1982 Atlantic City, NJ, U.S.A.	**1982 Pittsburgh Conference and Exhibition on Analytical Chemistry and Applied Spectroscopy** Contact: Mrs. Linda Briggs, Program Secretary, Pittsburgh Conference, Department J-057, 437 Donald Road, Pittsburgh, PA 15235, U.S.A. (Further details published in Vol. 212, No. 2)
March 28–April 2, 1982 Las Vegas, NV, U.S.A.	**183rd American Chemical Society National Meeting** Contact: A.T. Winstead, American Chemical Society, 1155 Sixteenth Street, NW, Washington, DC 20036, U.S.A.
April 5–8, 1982 Las Vegas, NV, U.S.A.	**International Symposium "Advances in Chromatography"** Contact: Prof. A. Zlatkis, Chemistry Department, University of Houston, Central Campus, 4800 Calhoun, Houston, TX 77004, U.S.A. Tel.: (713) 749-2623. (Further details published in Vol. 212, No. 3)
April 14–16, 1982 Amsterdam, The Netherlands	**12th Annual Symposium on the Analytical Chemistry of Pollutants** Contact: Prof. Dr. Roland W. Frei, The Free University, De Boelelaan 1083, 1081 HV Amsterdam, The Netherlands. (Further details published in Vol. 206, No. 1)
April 15–17, 1982 Tokyo, Japan	**International Symposium "Advances in Chromatography"** Contact: Prof. A. Zlatkis, Chemistry Department, University of Houston, Central Campus, 4800 Calhoun, Houston, TX 77004, U.S.A. Tel.: (713) 749-2623.
April 19–22, 1982 Barcelona, Spain	**International Congress on Automation in Clinical Laboratory** Contact: Dr. R. Galimany, Sección de Automatización, Laboratorio de Analisis Clinicos, C.S. "Principes de Espana", Hospitalet de Llobregat, Barcelona, Spain.
April 20th, 1982 Southend, Great Britain	**Analytical Problems in the Analysis of Agricultural Chemicals** Contact: The Royal Society of Chemistry, Analytical Division (East Anglia Region), Burlington House, London W1V 0BN, Great Britain.
April 20th, 1982 Belfast, Northern Ireland, U.K.	**Derivative Spectroscopy and its Applications in Bioanalytical and Environmental Chemistry** Contact: The Royal Society of Chemistry, Analytical Division (Northern Ireland Region), Burlington House, London W1V 0BN, Great Britain.
April 21–23, 1982 Neuherberg near Munich, G.F.R.	**Second International Workshop on Trace Element Analytical Chemistry in Medicine and Biology** Contact: Dr. P. Schramel, Gesellschaft fuer Strahlen- und Umweltforschung, Institut fuer Angewandte Physik, Physikalisch-Technische Abteilung, Ingolstaedter Landstrasse 1, D-8042 Neuherberg, G.F.R.
April 27–30, 1982 Munich, G.F.R.	**Biochemische Analytik 82** Contact: Prof. I. Trautschold, Medizinische Hochschule Hannover, Karl-Wiechert-Allee 9, 3000 Hannover 61, G.F.R. (Further details published in Vol. 224, No. 3)
May 2–6, 1982 Interlaken, Switzerland	**2nd International Symposium on Instrumental TLC (HPTLC)** Contact: Dr. R.E. Kaiser, Institute for Chromatography, P.O. Box 1141, D-6702 Bad Dürkheim, G.F.R.
May 11–14, 1982 Ghent, Belgium	**4th International Symposium on Quantitative Mass Spectrometry in Life Sciences** Contact: Prof. A. De Leenheer, Symposium Chairman, Laboratoria voor Medische Biochemie en voor Klinische Analyse, De Pintelaan 135, B-9000 Ghent, Belgium.

June 6–12, 1982 Frankfurt, G.F.R.	**European Meeting on Chemical Engineering and ACHEMA Exhibition Congress 1982** Contact: DECHEMA P.O. Box 970146, D-6000 Frankfurt/M 97, G.F.R.
June 7–11, 1982 Philadelphia, PA, U.S.A.	**VI International Symposium on Column Liquid Chromatography** Contact: R.A. Barford, ERRC – SEA, U.S. Department of Agriculture, 600 E. Mermaid Lane, Philadelphia, PA 19118, U.S.A. (Further details published in Vol. 211, No. 3)
June 21–23, 1982 Bordighera (near San Remo), Italy	**International Symposium on Chromatography and Mass Spectrometry in Biomedical Sciences** Contact: Dr. Alberto Frigerio, c/o Istituto di Ricerche Farmacologiche "Mario Negri", Via Eritrea, 62 – 20157 Milan, Italy. Tel.: 35.54.546; Telex: 331268 NEGRI I.
June 28–30, 1982 East Lansing, MI, U.S.A.	**35th American Chemical Society Annual Summer Symposium** Contact: A.I. Popov, Chemistry Department, Michigan State University, East Lansing, MI 48824, U.S.A.
July 11–16, 1982 Washington, DC, U.S.A.	**6th International Conference on Computers in Chemical Research and Education (ICCCRE)** Contact: Dr. Stephen R. Heller, Chairman, 6th ICCCRE, EPA, MIDSD, PM–218, 401 M Street, S.W., Washington, DC 20460, U.S.A. Tel: (202) 755–4938, Telex: 89–27–58.
July 11–16, 1982 Louvain-la-Neuve, Belgium	**6th IUPAC Conference on Physical Organic Chemistry** Contact: Prof. A, Bruylants, Université Catholique de Louvain, Laboratoire de Chimie Generale et Organique, Batiment Lavoisier 1 Place Louis Pasteur, 1348 Louvain-la-Neuve, Belgium.
July 12–16, 1982 Amherst, MA, U.S.A.	**IUPAC Macromolecular Symposium** Contact: James C.W. Chien, Department of Polymer Science & Engineering, University of Massachusetts, Amherst, MA 01003, U.S.A.
July 19–22, 1982 Prague, Czechoslovakia	**Prague Microsymposium "Selective Sorbents"** Contact: Dr. F. Svec, c/o Institute of Macromolecular Chemistry, Czechoslovak Academy of Sciences, Heyrovského n.2, 162 06 Prague, Czechoslovakia.
Aug. 11–13, 1982 Hameenlinna, Finland	**6th European Symposium on Polymer Spectroscopy (ESOPS 6)** Contact: Professor Johan Lindberg, Department of Wood and Polymer Chemistry, University of Helsinki, Meritullinkatu 1 A, SF 00170 Helsinki 17, Finland.
Aug. 15–21, 1982 Perth, Australia	**The 12th International Congress of Biochemistry** Contact: Brian Thorpe, Department of Biochemistry, Faculty of Science Australian National University, Canberra A.C.T. 2600, Australia.
Aug. 23–27, 1982 Budapest, Hungary	**22nd International Conference on Coordination Chemistry** Contact: Prof. M.T. Beck, Institute of Physical Chemistry, Kossuth Lajos University, Debrecen 10, H–4010, Hungary.

Aug. 29–Sept. 4, 1982 Kyoto, Japan	**5th International Congress of Pesticide Chemistry** Contact: Rikagaku Kenysho (The Institute of Physical and Chemical Research), 2–1 Hirosawa Wako-shi Saitama Pref. 351, Japan.
Aug. 30–Sep. 3, 1982 Vienna, Austria	**9th International Mass Spectrometry Conference** Contact: Interconvention, P.O. Box 105, A–1014 Vienna, Austria. (Further details published in Vol. 206, No. 1)
Aug. 31–Sept. 2, 1982 Vienna, Austria	**5th International IUPAC Symposium on Mycotoxins and Phycotoxins** Contact: Prof. P. Krogh, Department of Veterinary Microbiology, Purdue University, West Lafayette, IN 47907, U.S.A.
Sept. 5–9, 1982 Liĕge, Belgium	**Eighth European Workshop on Drug Metabolism** Contact: Professor Jacques E. Gielen, Laboratoire de Chimie Médicale, Institut de Pathologie, Université de Liĕge, Bâtiment B 23, B-4000 Sart Tilman par Liĕge 1, Belgium. Tel.: (32-41)-56.24.80/81.
Sept. 7–11, 1982 Hradec Králové, Czechoslovakia	**8th International Symposium on Biomedical Applications of Chromatography** Contact: Dr. K. Macek, Institute of Physiology, Czechoslovakian Academy of Sciences, Vídeňská 1083, CS-142 20, Prague 4-Krč, Czechoslovakia.
Sept. 12–17, 1982 Kansas City, MO, U.S.A.	**184th American Chemical Society National Meeting** Contact: A.T. Winstead, American Chemical Society, 1155 Sixteenth Street, NW, Washington DC 20036, U.S.A.
Sept. 13–17, 1982 London, Great Britain	**14th International Symposium on Chromatography** Contact: The Executive Secretary, Chromatography Discussion Group, Trent Polytechnic, Burton Street, Nottingham, NG1 4BU, Great Britain. (Further details published in Vol. 211, No. 3)
Sept. 19–24, 1982 Philadelphia, PA, U.S.A.	**9th National Meeting of the Federation of Analytical Chemistry and Spectroscopy Societies (FACSS)** Contact: Division of Analytical Chemistry, American Chemical Society, Department of Chemistry, Notre Dame, IN 46556, U.S.A.
May 30–June 3, 1983 Melbourne, Australia	**International Conference on Chromatographic Detectors** Contact: The Secretary, International Conference on Chromatographic Detectors, University of Melbourne, Parkville, Victoria 3052, Australia.
July 17–23, 1983 Edinburgh, Scotland, Great Britain	**SAC '83, International Conference and Exhibition on Analytical Chemistry** Contact: The Royal Society of Chemistry, Analytical Division, Burlington House, London W1V 0BN, Great Britain. Tel.: 01-734-9971.
Aug. 28–Sept. 2, 1983 Amsterdam, The Netherlands	**9th International Symposium on Microchemical Techniques** Contact: Symposium Secretariat, c/o Municipal Congress Bureau, Oudezijds Achterburgwal 199, 1012 DK Amsterdam, The Netherlands. Tel: (020) 552 3459.

PUBLICATION SCHEDULE FOR 1981

Journal of Chromatography (incorporating *Chromatographic Reviews*) and *Journal of Chromatography, Biomedical Applications*

MONTH	N 1980	D 1980	J	F	M	A	M	J	J	A	S	O	N	D
Journal of Chromatography			203 204 205/1 205/2	206/1 206/2 206/3	207/1 207/2 207/3	208/1 208/2 209/1	209/2 209/3 210/1	210/2 210/3 211/1	211/2 211/3 212/1 212/2	212/3 213/1 213/2	213/3 214/1 214/2	214/3 215 216	217 218 219/1	219/2 219/3
Chromatographic Reviews							220/1					220/2		220/3
Biomedical Applications	221/1	221/2	222/1	222/2	222/3	223/1	223/2	224/1	224/2	224/3	225/1	225/2	226/1	226/2

INFORMATION FOR AUTHORS

(Detailed *Instructions to Authors* were published in Vol. 209, No. 3, pp. 501–504. A free reprint can be obtained by application to the publisher)

Types of Contributions. The following types of papers are published in the *Journal of Chromatography* and the section on *Biomedical Applications*: Regular research papers (Full-length papers), Short communications and Notes. Short communications are preliminary announcements of important new developments and will, whenever possible, be published with maximum speed. Notes are usually descriptions of short investigations and reflect the same quality of research as Full-length papers, but should preferably not exceed four printed pages. For reviews, see page 2 of cover under Submission of Papers.

Submission. Every paper must be accompanied by a letter from the senior author, stating that he is submitting the paper for publication in the *Journal of Chromatography*. Please do not send a letter signed by the director of the institute or the professor unless he is one of the authors.

Manuscripts. Manuscripts should be typed in double spacing on consecutively numbered pages of uniform size. The manuscript should be preceded by a sheet of manuscript paper carrying the title of the paper and the name and full postal address of the person to whom the proofs are to be sent. Authors of papers in French or German are requested to supply an English translation of the title of the paper. As a rule, papers should be divided into sections, headed by a caption (*e.g.*, Summary, Introduction, Experimental, Results, Discussion, etc.). All illustrations, photographs, tables, etc. should be on separate sheets.

Introduction. Every paper must have a concise introduction mentioning what has been done before on the topic described, and stating clearly what is new in the paper now submitted.

Summary. Full-length papers and Review articles should have a summary of 50–100 words which clearly and briefly indicates what is new, different and significant. In the case of French or German articles an additional summary in English, headed by an English translation of the title, should also be provided. (Short communications and Notes are published without a summary.)

Illustrations. The figures should be submitted in a form suitable for reproduction, drawn in Indian ink on drawing or tracing paper. Each illustration should have a legend, all the *legends* being typed (with double spacing) together on a *separate sheet*. If structures are given in the text, the original drawings should be supplied. Coloured illustrations are reproduced at the author's expense, the cost being determined by the number of pages and by the number of colours needed. The written permission of the author and publisher must be obtained for the use of any figure already published. Its source must be indicated in the legend.

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Modern Thin-Layer Chromatography

can only be fully exploited with a complete system of instruments for sample application, chromatographic development and evaluation

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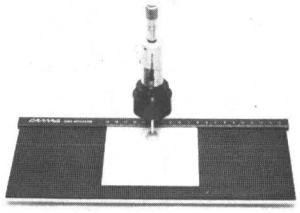
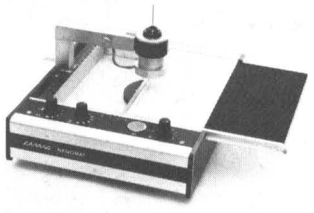
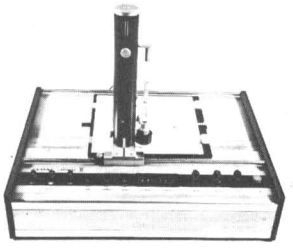
1. Sample Application



CAMAG Nano-Applicator
variable sample volumes 50–230 nl (spotwise), reproducibility 1%

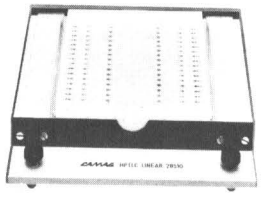


or **CAMAG Nanomat**
fixed sample volumes 100 or 200 nl with nanopipette, or larger volumes using the automatic repetition device



or **CAMAG Linomat III**
applies dilute sample solutions (2–100 µl) in form of a narrow band; reproducibility 1%

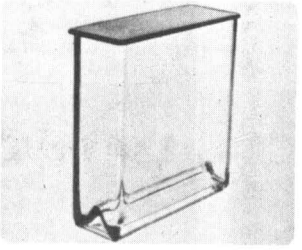
2. Chromatogram Development



CAMAG Twin-Trough Chamber ▶
versatile, economical in solvent consumption

or

◀ **CAMAG HPTLC Linear Developing Chamber**
up to 32 samples can be separated on one 10 x 10 cm HPTLC-plate or 72 samples on a 20 x 10 cm plate



3. Quantitative Evaluation

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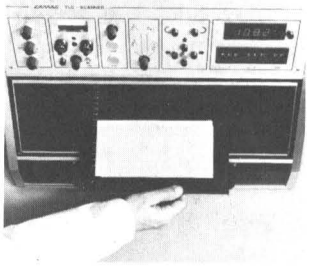
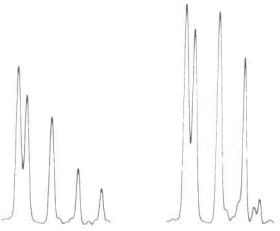
Scanning modes: absorbance, fluorescence, fluorescence quenching.
Automatic scanning of all tracks of a chromatogram, or of the selected chromatogram segment.





If you are planning to set up a new TLC laboratory, or to complement your existing TLC equipment, ask the CAMAG specialist. He will help.

Ask for full information on COMPLETE CAMAG SYSTEMS for circular HPTLC and for conventional TLC also.

CAMAG

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