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

MONTH	D 1979	J	F	M	A	M	J	J	A	S	O	N	D
<i>Journal of Chromatography</i>	185 186	187/1 187/2 188/1	188/2 189/1 189/2	189/3 190/1	190/2 191 192/1	192/2 193/1 193/2 193/3	The publication schedule for further issues will be published later.						
<i>Chromatographic Reviews</i>			184/1 184/2										
<i>Biomedical Applications</i>		181/1 181/2		181/ 3-4	182/1	182/2							

Scope. The *Journal of Chromatography* publishes papers on all aspects of chromatography, electrophoresis and related methods. Contributions consist mainly of research papers dealing with chromatographic theory, instrumental development and their applications. The section *Biomedical Applications*, which is under separate editorship, deals with the following aspects: developments in and applications of chromatographic and electrophoretic techniques related to clinical diagnosis (including the publication of normal values); screening and profiling procedures with special reference to metabolic disorders; results from basic medical research with direct consequences in clinical practice; combinations of chromatographic and electrophoretic methods with other physico-chemical techniques such as mass spectrometry. In *Chromatographic Reviews*, reviews on all aspects of chromatography, electrophoresis and related methods are published.

Submission of Papers. Papers in English, French and German may be submitted, if possible in three copies. Manuscripts should be submitted to:

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ELECTROANALYSIS IN HYGIENE, ENVIRONMENTAL, CLINICAL and PHARMACEUTICAL CHEMISTRY

Proceedings of a Conference organised by the Electroanalytical Group of the Chemical Society, London, held at Chelsea College, University of London, U.K., 17-20 April, 1979.

edited by W. FRANKLIN SMYTH, Chelsea College, University of London, U.K.

ANALYTICAL CHEMISTRY SYMPOSIA SERIES 2

The proceedings of this international conference comprise 39 papers, principally concerned with how potentiometric and voltammetric methods of electroanalysis are used to solve a wide range of analytical problems in the fields of clinical chemistry, hygiene, pharmacy, pharmacology and environmental chemistry.

Although the papers reflect the many topics under discussion certain themes predominate. These include investigation of the actual electrochemical techniques and instrumentation, the importance of the mechanism of the electrochemical reaction at the chosen indicator electrode in optimising the electroanalytical 'finish', and direct, rapid and sensitive measurement in complex biological matrices. The results on the determination of inorganic, organic and organometallic substances in complex matrices such as body fluids, factory air and the aqueous environment are also discussed. On-line analysis with and without a prior separation process, speciation studies by the application of 'cold' electroanalytical methods and details of novel electrode construction, instrumental design and analytical methods complete the discussion.

SELECTED CONTENTS: Plenary Lectures. New Ion Selective Electrodes and Their Clinical and Biological Application (D. Amman, H.-B. Jenny, P. C. Meier and W. Simon). Polarographic Analysis of Nucleic Acids (E. Palaček). Electrochemical Gas Monitors in Occupational Hygiene (I. Bergman). Electroanalytical Applications in Pharmacy and Pharmacology (G. J. Patriarche and J.-C. Vire). Stripping Voltammetry of Molecules of Pharmaceutical Importance (W. Franklin Smyth). A Critical Assessment of the Voltammetric Approach for the Study of Toxic Trace Metals in Biological Specimens (H. W. Nürnberg). Polarographic Analysis of Pesticides in Food Products (J. Davidék). **Keynote Lectures.** Design Principles and Behaviour of Sensitive Calcium Ion-Selective Electrodes (G. J. Moody and J. D. R. Thomas). The Determination of Radiation Damages in Native DNA by Single Sweep Voltammetry (J. M. Sequaris and P. Valenta). The Importance of Measuring Oxidation-Reduction Systems in Clinical Research (J. Chayen). Determination of Mercury in Urine by Potentiometric Stripping Analysis (D. Jagner and K. Árén). Differential Pulse Polarographic Determination of Drugs in Pharmaceutical Formulations (E. Jacobsen). Trace Level Polarographic Analysis of Drugs in Body Fluids (M. A. Brooks). Electrochemical Approaches to Environmental Pollution Control (S. das Gupta, B. Fleet and I. F. T. Kennedy). Electroanalysis of Economic Poisons (J. Osteryoung, J. W. Whitaker and M. R. Smyth).

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ELECTROPHORESIS

A SURVEY OF TECHNIQUES AND APPLICATIONS

Part A: Techniques

Z. DEYL, Czechoslovak Academy of Sciences, Prague (editor)
F. M. EVERAERTS, Z. PRUSÍK, and P. J. SVENDSEN (co-editors)

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This first volume in a two part set, deals with the principles, theory and instrumentation of modern electromigration methods. The second volume will be concerned with details of applications of electromigration methods to diverse categories of compounds, although a few applications are already discussed in Part A.

Some electromigration methods have become standard procedures because of their extensive use in analytical and preparative separations. These are discussed together with newer developments in the field. Hints are included to help the reader to overcome difficulties frequently arising from the lack of suitable equipment. Adequate theoretical background of the individual techniques is included. A theoretical approach to the deteriorative processes is presented in order to facilitate further development of a particular technique and its application to a special problem.

In each chapter practical realizations of different techniques are discussed and examples are presented to demonstrate the limits of each method. The mathematical and physicochemical background is arranged so as to make it as coherent as possible for both non-professionals such as post-graduate students, and experts using electromigration techniques.

CONTENTS: Preface. Foreword. Introduction. Chapters: 1. Theory of electromigration processes. 2. Classification of electromigration methods. 3. Evaluation of the results of electrophoretic separation. 4. Molecular size and shape in electrophoresis. 5. Zone electrophoresis (except gel-type techniques and immunoelectrophoresis). 6. Gel-type techniques. 7. Quantitative immunoelectrophoresis. 8. Moving boundary electrophoresis in narrow-bore tubes. 9. Isoelectric focusing. 10. Analytical isotachopheresis. 11. Continuous flow-through electrophoresis. 12. Continuous flow deviation electrophoresis. 13. Preparative electrophoresis in gel-media. 14. Preparative electrophoresis in columns. 15. Preparative isoelectric focusing. 16. Preparative isotachopheresis. 17. Preparative isotachopheresis on the micro scale. List of frequently occurring symbols. Subject index.

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Obituary

Professor K. V. Chmutov (1902-1979)

The prominent Soviet scientist Konstantin Vasilyevich Chmutov, Chairman of the Scientific Council on Chromatography of the U.S.S.R. Academy of Sciences, Corresponding Member of the U.S.S.R. Academy of Sciences, died on May 29th, 1979.

His first scientific paper was published as far back as 1925 when he was a student in the Chemistry Department at the Technical High School in Moscow.



Under the sponsorship and supervision of the famous Russian scientist N. A. Shilov, he conducted research into the interaction of solute ions with surface oxides of carbon and proposed the existence of three kinds of surface oxides exhibiting basic, amphoteric and acidic properties. This theory contributed to the development of ion-exchange chromatography.

A wealth of data on the statics, kinetics and dynamics of sorption were presented by K. V. Chmutov together with M. M. Dubinin in their well known book *The Physicochemical Fundamentals of Gas Masks*.

In 1950, K. V. Chmutov set up a chromatographic laboratory at the Institute of Physical Chemistry, U.S.S.R. Academy of Sciences, in which fundamental studies on sorption in ion-exchange and molecular chromatography were carried out. These studies have made it possible to improve substantially industrial chromatographic processes through the use of new techniques and instrumentation.

K. V. Chmutov was a brilliant experimenter. His skill in providing elegant and simple solutions to involved experimental problems was remarkable. His book *The Techniques of Physicochemical Research* has become a handbook for Soviet physical chemists. For many years, K. V. Chmutov was Deputy Editor-in-Chief of *Zhurnal Fizicheskoi Khimii* and a member of the Editorial Board of the *Journal of Chromatography*. He has written and edited a great number of papers and books.

K. V. Chmutov was Chairman of the Scientific Council on Chromatography since its inception and made a major contribution to the development of chromatography in the Soviet Union as well as of cooperation between chromatographers in the U.S.S.R. and the rest of the world.

K. V. Chmutov was a man of versatile erudition, a connoisseur of fine arts and letters. His conversation was a cascade of wit, and his colleagues often became innocent targets of ingenious epigrams and playful caricatures. He was a man of noble ideals and generous heart who always treated people with compassion.

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CHROM. 12,442

NEW RESULTS WITH A FLOW-IMPEDANCE BRIDGE DETECTOR

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(Received August 10th, 1979)

SUMMARY

A new version of a pneumatic analogue of the Wheatstone bridge as a gas chromatographic detector is described. The detector response is defined in terms of the viscosity difference between the carrier gas and the mixture of carrier gas and solute, and the volume of this gas mixture. A relationship is derived between the measured surface area and the amount of pure solute injected.

INTRODUCTION

In a previous paper¹ we reported the results of some preliminary experiments with a new version of a flow-impedance bridge detector for gas chromatography (GC)^{2,3}. This detector is based on the dependence of the flow resistance of a capillary on the nature of the gas. Four equal capillaries are circuited in a Wheatstone-like bridge. A flow diagram of the detector is shown in Fig. 1.

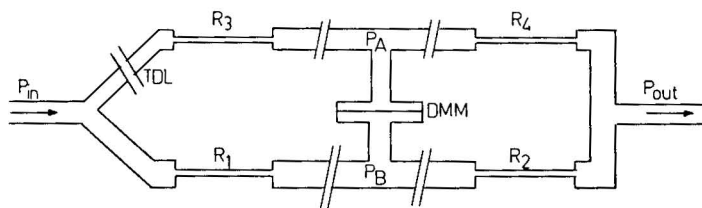


Fig. 1. Flow diagram of the detector.

R_1 , R_2 , R_3 and R_4 are the capillaries and P_{in} and P_{out} are the pressures at the inlet and outlet of the detector, respectively. TDL represents the time delay line and DMM the differential manometer that measures the difference between the pressures P_A and P_B . A single injection of a solute-carrier gas mixture into the flow of carrier gas through the detector results in four peaks in the output. These four peaks correspond to the passage at different times of the gas mixture through the capillaries. This shift is obtained with the time delay line. Using the experimental data collected with this prototype, a new apparatus has been developed, in the response of which

only the first of the four peaks remains. This was achieved by increasing all over the detector the effects of mixing and diffusion, except for the connection between the detector inlet and the capillary R_1 , which is made smooth in order to maintain a sharp first peak.

In this paper we describe the properties of the detector and present some results that have been obtained.

DETECTOR RESPONSE

We shall describe the response of the detector upon a single injection of a solute-carrier gas mixture, resulting in a variation of the viscosity at the inlet of the detector, as shown in Fig. 2. The time interval t_m in Fig. 2 satisfies the equation

$$t_m = \frac{V_m}{\Phi_D} \quad (1)$$

where V_m represents the volume of the gas mixture injected and Φ_D the flow-rate of



Fig. 2. Viscosity variation at the inlet of the detector as a function of time.

carrier gas through the detector. Throughout we shall restrict the discussion to those situations where four restrictions hold:

$$(i) P_{in} - P_{out} \ll P_0$$

with

$$P_0 = \frac{P_{in} + P_{out}}{2}$$

$$(ii) \eta_m - \eta_c = \eta_d \ll \eta_c$$

where η_m and η_c are the viscosities of the gas mixture and carrier gas, respectively;

(iii) the time interval t_{cap} needed for the front of the gas mixture to pass a capillary is small compared with all time intervals used in the calculations; and

(iv) the Poiseuille equation can be used in the following way:

$$\Phi_{cap} = \frac{\alpha}{\eta} \cdot P_{cap} \quad (2)$$

with $\alpha = \pi r^4/8l$, where Φ_{cap} is the flow through and P_{cap} the pressure drop across a capillary of radius r and length l .

In the calculations of the response we require the specifications of the differential manometer. As the two compartments of the manometer are separated by a flexible membrane, the two volumes of the compartments will be dependent on the pressure difference between them. The volumes of compartments A and B can be expressed by the equations

$$V_A = V_0 + \beta(P_A - P_B) \quad (3a)$$

$$V_B = V_0 - \beta(P_A - P_B) \quad (3b)$$

where β is the compliance of the membrane of the manometer ($\beta = \partial V / \partial P$). V_0 includes those volumes which are directly connected to the compartments.

The pressure difference, $P_A - P_B$, caused by the injection can be expressed by

$$P_A - P_B = -\frac{\eta_d}{\eta_c} \cdot \frac{P_{in} - P_{out}}{4} \cdot (1 - e^{-t/\tau}) \quad (4)$$

with

$$\tau = \frac{\eta_c V_0}{2\alpha P_0} \left(1 + \frac{2\beta P_0}{V_0}\right)$$

where $t = 0$ is the time when the front of the gas mixture arrives at the capillary R_1 . For $t > t_m$ the pressure difference $P_A - P_B$ decreases exponentially to zero. Assuming $t_m > \tau$ we can describe the detector response by (see Fig. 3)

$$P_A - P_B = -\frac{\eta_d}{\eta_c} \cdot \frac{P_{in} - P_{out}}{4} \cdot (1 - e^{-t/\tau}) \quad 0 \leq t \leq t_m \quad (5a)$$

$$P_A - P_B = -\frac{\eta_d}{\eta_c} \cdot \frac{P_{in} - P_{out}}{4} \cdot e^{-(t-t_m)/\tau} \quad t > t_m \quad (5b)$$

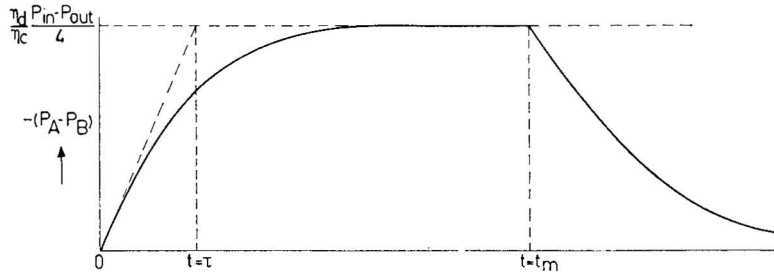


Fig. 3. Detector response on an injection, assuming $t_m > \tau$.

Another situation where we can easily calculate the detector response is characterized by $t_m \ll \tau$. We can then use the linear approximation of eqn. 5a, giving (see Fig. 4)

$$P_A - P_B = -\frac{\eta_d}{\eta_c} \cdot \frac{P_{in} - P_{out}}{4} \cdot \frac{t}{\tau} \quad 0 \leq t \leq t_m \quad (6a)$$

$$P_A - P_B = -\frac{\eta_d}{\eta_c} \cdot \frac{P_{in} - P_{out}}{4} \cdot \frac{t_m}{\tau} \cdot e^{-(t-t_m)/\tau} \quad t \geq t_m \quad (6b)$$

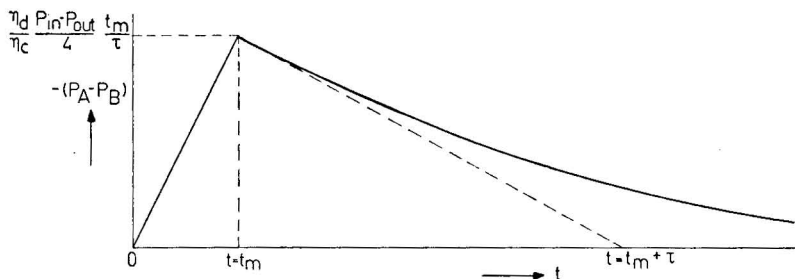


Fig. 4. Detector response on an injection, assuming $t_m \ll \tau$.

Considering Fig. 3, it will be of interest to calculate the surface area, S , of the peak. With the additional condition $t_m \gg \tau$ we shall relate this surface area to the volume, V_s , of pure solute (viscosity η_s) in the injected gas mixture. We further assume a linear relationship between η_d and the concentration of solute in the carrier gas:

$$\eta_d V_m = (\eta_m - \eta_c) V_m = (\eta_s - \eta_c) V_s \quad (7)$$

For the surface area, S , we obtain

$$S = \int_0^\infty (P_A - P_B) dt = (P_A - P_B) t_m = - \frac{\eta_d}{\eta_c} \cdot \frac{P_{in} - P_{out}}{4} \cdot t_m \quad (8)$$

Making use of eqns. 1, 2 and 7, we have

$$S = - \frac{\eta_d V_m}{4\alpha} = - \frac{\eta_s - \eta_c}{4\alpha} \cdot V_s \quad (9)$$

Eqn. 9 indicates that the detector can be used for quantitative purposes. If we know the values of the viscosities η_s of the pure solute and η_c of the carrier gas at pressure P_0 , the measured value of the surface area, S , leads to the volume, V_s , that the pure solute will have at the same pressure. It should be noticed that with a non-linear relationship between the composition and viscosity of the gas mixture, corrections are necessary.

To ascertain the detection limit, ΔV_s , of the pure solute, we start with the uncertainty, ΔS , of the surface area caused by the manometer noise, ΔP :

$$\Delta S = t_m \Delta P \quad (10)$$

Using eqn. 9 and taking the signal-to-noise ratio as $S/N = 1$, we obtain

$$\Delta V_s = \frac{4\alpha}{\eta_s - \eta_c} \cdot t_m \Delta P \quad (11)$$

For the purpose of estimation, we use $t_m = \tau$ and obtain

$$\Delta V_s = \frac{2\eta_c}{\eta_s - \eta_c} \left(1 + \frac{2\beta P_0}{V_0}\right) V_0 \cdot \frac{\Delta P}{P_0} \quad (12)$$

The same result can be obtained by using eqn. 6a with $t = t_m$ and $S/N = 1$.

EXPERIMENTAL

The capillaries of the bridge are made of stainless steel with an I.D. of 0.038 cm and a length of 10.75 cm, leading to $\alpha = (4.76 \pm 0.04) \cdot 10^{-9} \text{ cm}^3$. The time delay line consists of a nylon tube with an I.D. of 0.4 cm and a length of 100 cm. This type of tube is also used to connect R_1 with R_2 and R_3 with R_4 , both connections having a length of 50 cm. As the volume of a manometer compartment is about 1 cm^3 , we have $V_0 = 7.3 \text{ cm}^3$. For the compliance of the manometer membrane we use $\beta = 1.1 \cdot 10^{-2} \text{ cm}^3/\text{torr}$. P_{out} is atmospheric pressure and $P_{\text{in}} - P_{\text{out}}$ has a maximum value of 1 torr, so $P_0 = 760 \text{ torr}$. The values of the time constant, τ , of the detector are 0.44 and 0.39 sec with nitrogen and helium, respectively, as the carrier gas. Three different systems are used for injecting the solute into the flow of carrier gas (see Fig. 5):

- (i) syringe, $V_{\text{inj}} \leq 50 \mu\text{l}$ (Fig. 5a);
- (ii) injection valve, $230 \mu\text{l} \leq V_{\text{loop}} \leq 1120 \mu\text{l}$ (Fig. 5b);
- (iii) syringe in combination with a splitter (Fig. 5c); division factor $K = \Phi_D / (\Phi_D + \Phi_R)$.

All of the experiments were performed at room temperature.

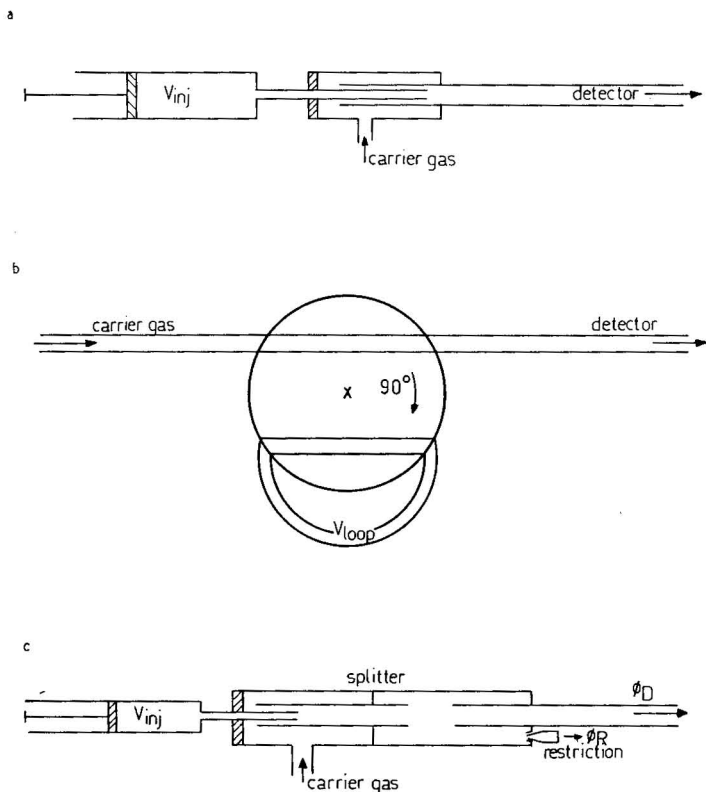
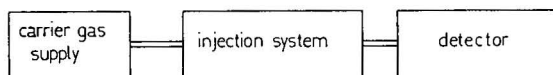


Fig. 5. Injection systems used in the experiments.

RESULTS AND DISCUSSION

Experiments were performed with nitrogen as carrier gas and oxygen as solute. This combination of gases was chosen because of the fairly linear relationship between the concentration of oxygen in nitrogen and the viscosity of the mixture⁴. Injection systems (i) and (ii) were used and directly coupled to the detector (see Fig. 6a). In Fig. 7, the measured values of the surface area under the peak are

a



b

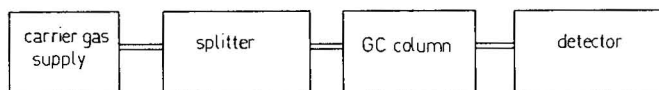


Fig. 6. Schematic illustration of the experimental arrangement.

shown as a function of the volume of oxygen injected. The volume V_v in Fig. 7b represents the dead space of the injection valve. The slope of the lines in Fig. 7 is

$$\left(\frac{dS}{dV_s}\right)_{\text{exp}} = -(1.11 \pm 0.01) \cdot 10^{-3} \text{ torr} \cdot \text{sec}/\mu\text{l} \quad (13)$$

Calculating this slope with eqn. 9 we obtain

$$\left(\frac{dS}{dV_s}\right)_{\text{th}} = -\frac{\eta_s - \eta_c}{4\alpha} \quad (14)$$

Substitution of $\eta_s = \eta_{\text{O}_2} = (1.528 \pm 0.006) \cdot 10^{-7} \text{ torr} \cdot \text{sec}$ and $\eta_c = \eta_{\text{N}_2} = (1.322 \pm 0.006) \cdot 10^{-7} \text{ torr} \cdot \text{sec}$ leads to

$$\left(\frac{dS}{dV_s}\right)_{\text{th}} = -(1.08 \pm 0.07) \cdot 10^{-3} \text{ torr} \cdot \text{sec}/\mu\text{l} \quad (15)$$

The agreement with the experimental value is good.

To test the detector in combination with a gas chromatographic column, the arrangement shown in Fig. 6b was applied. The splitter in this arrangement enables

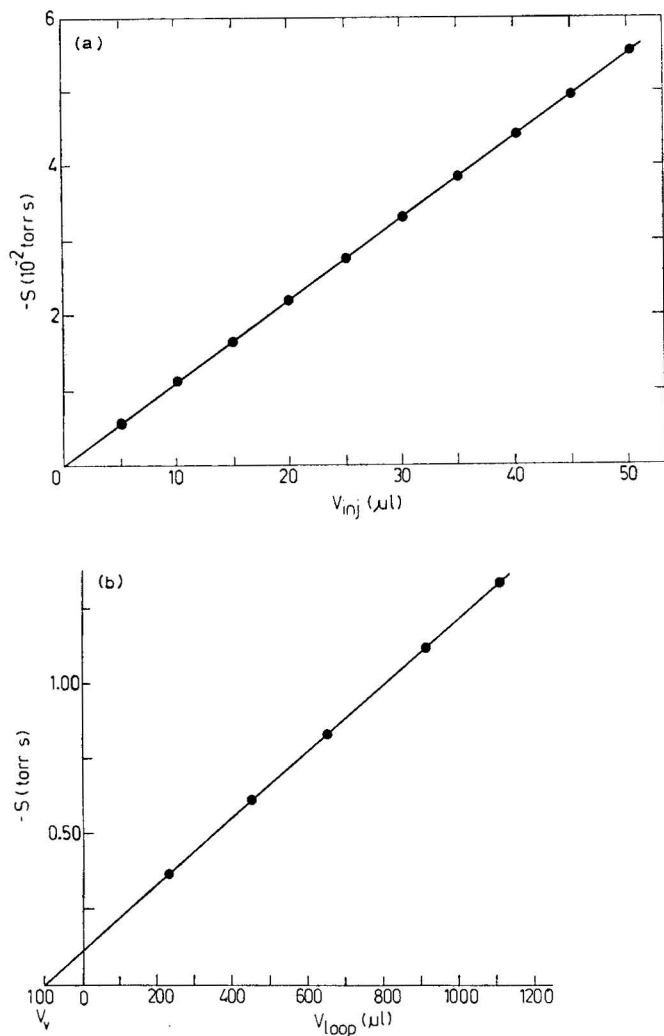


Fig. 7. Plots of the measured surface area of oxygen against the volume of oxygen injected, with nitrogen as carrier gas. $\Phi_D = 10^{-2}$ ml/sec.

us to inject the column solute volumes, V_s , that are much smaller than the syringe volumes, V_{inj} :

$$V_s = KV_{inj} \quad (16)$$

where $K \approx 10^{-2}$.

Helium was used as the carrier gas and natural gas as the solute and the GC column (50 m $\times$ 0.02 cm I.D.) contained squalane. As this work was not concerned with optimization of the GC column but rather the exploitation of the detection principle, optimal conditions from the viewpoint of column efficiency were not sought.

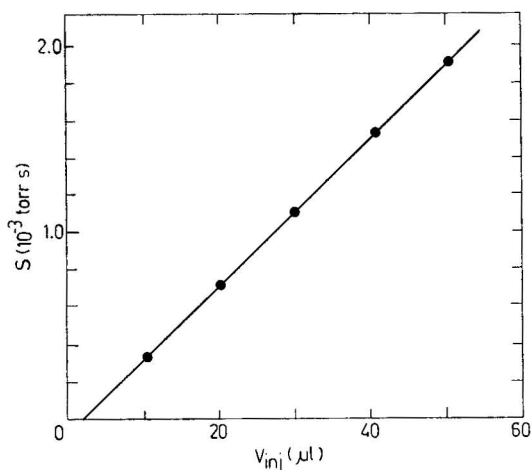


Fig. 8. Plot of surface area of natural gas against the volume injected. Conditions: helium as carrier gas; $K = 7.4 \cdot 10^{-3}$; $\Phi_p = 10^{-2}$ ml/sec.

In Fig. 8 the measured values of the surface area are plotted against the volumes of natural gas injected. The fact that the line in Fig. 8 does not pass through the origin is due to the dead space in the syringe, which has to be taken into account because of the excess pressure (2.5 atm) in the splitter. The slope of the line through the measured points satisfies the equation

$$\left(\frac{dS}{dV_{inj}} \right)_{exp} = (3.93 \pm 0.05) \cdot 10^{-5} \text{ torr} \cdot \text{sec}/\mu\text{l} \quad (17)$$

According to eqns. 9 and 16, this slope is given by

$$\left(\frac{dS}{dV_{inj}} \right)_{th} = - \frac{\eta_s - \eta_c}{4a} \cdot K \quad (18)$$

where $K = 7.4 \cdot 10^{-3}$. To be able to compare this with the experimental value, we need to know the value of $\eta_s - \eta_c$. Because of the non-linear relationship between the viscosity and the concentration of natural gas in helium, we cannot use the difference between the viscosities of the pure gases. As the concentrations of natural gas that are found in the detector are below 2%, we use the linear extrapolation of the low concentration data, resulting in $\eta_s - \eta_c = -(1.06 \pm 0.03) \cdot 10^{-7}$ torr·sec. Substitution of this value in eqn. 18 leads to

$$\left(\frac{dS}{dV_{inj}} \right)_{th} = (4.1 \pm 0.2) \cdot 10^{-5} \text{ torr} \cdot \text{sec}/\mu\text{l} \quad (19)$$

This is in good agreement with the experimental value.

To estimate the experimental detection limit, ΔV_s , the measured response from an injection of 10 μl of natural gas in helium is shown in Fig. 9, which corresponds with the experiment at the lowest value of V_{inj} as shown in Fig. 8. From Fig. 9 we can estimate the noise, ΔP , of the manometer to be 10^{-5} torr. Owing

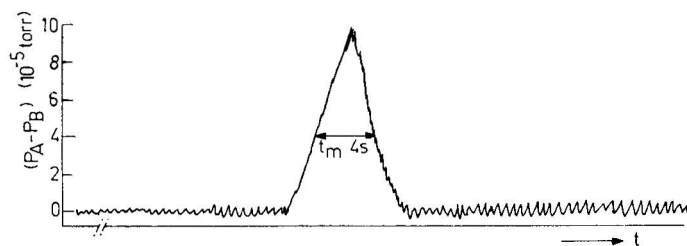


Fig. 9. Detector response on injection of $10\ \mu\text{l}$ of natural gas. Conditions: helium as carrier gas; $K = 7.4 \cdot 10^{-3}$; $\Phi_D = 10^{-2}\ \text{ml/sec}$.

to the splitter, only $0.08\ \mu\text{l}$ of the injected volume of $10\ \mu\text{l}$ reaches the detector. It can be seen in Fig. 9 that the peak is well above the noise level and that a gain of one order of magnitude is possible, giving $(\Delta V_s)_{\text{exp}} = 8 \cdot 10^{-3}\ \mu\text{l}$.

This value is also found when calculating ΔV_s with eqn. 11 and substitution of $t_m = 5\ \text{sec}$. According to eqn. 11, smaller values of ΔV_s should be possible when using smaller values of t_m . The limit of this gain is reached as $t_m = \tau$ and satisfies, according to eqn. 12, $(V_s)_{\text{th}} = 9 \cdot 10^{-4}\ \mu\text{l}$.

The chromatogram shown in Fig. 10 was obtained by injecting $60\ \mu\text{l}$ of a mixture containing air and hydrocarbons into a flow of helium, and using the arrangement shown in Fig. 6b.

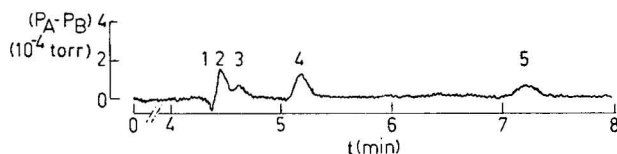


Fig. 10. Chromatogram obtained by injecting $60\ \mu\text{l}$ of a mixture containing air and hydrocarbons. Conditions: helium as carrier gas; $K = 10^{-2}$; $\Phi_D = 10^{-2}\ \text{ml/sec}$. Peaks: 1 = air; 2 = methane; 3 = ethane; 4 = propane; 5 = butane.

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SYNTHESIS AND GAS CHROMATOGRAPHIC USE OF METAL-CONTAINING SURFACES*

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SUMMARY

A variety of volatile metal halides were used in the gas phase to produce layers with thicknesses in the monomolecular range on Chromosorb W. The final layers result from subsequent treatment with H_2O , NH_3 or H_2 . They could be considered to be of the "oxide", "nitride" or "reduced" type, although no evidence for particular surface structures has been obtained. Layers were tested by gas chromatography directly and after attempted synthesis of an additional "bonded" Carbowax 20M layer. These procedures were successful (in terms of achieving inorganic layers in the monomolecular range and good chromatography with Carbowax 20M) in some but not all cases. The best materials were about equal in chromatographic performance to the best comparable phases containing no metals. "Bonding" of Carbowax could be achieved on several types of layers. For example, those containing Fe or Cr performed noticeably better than those containing Al or B. General uses of such materials are suggested.

INTRODUCTION

Metal compounds can play a role in, on, under or as chromatographic phases. If they are present by design, their effects are considered beneficial. This is perhaps clearest seen in complexation chromatography (for a review see ref. 1; some recent papers are cited as refs. 2–10). For this technique, metals are used in the form of salts, ions (on an ionexchange matrix) or chelates.

Somewhat different from this application is the role of metal oxides. Aside from silica (and, on occasion, alumina), they have but rarely served as gas chromato-

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graphic (GC) phases or supports. Hydrated ferric oxide has been used for the analysis of gases^{11,12} and so has chromia¹³. Ferric oxide has also carried thin films of Carbowax 20M¹⁴ and heavier loads of the liquid phase triethylene glycol¹⁵. Several other materials, *e.g.*, antimony pentoxide¹⁶, have also been investigated.

If metals are present on chromatographic supports owing to natural or, unwittingly, industrial processes, their influence is considered detrimental. The presumably macromolecular forms of iron and aluminum on the surface of diatomaceous earth supports may serve as the most common example.

To decrease their concentration, extraction with HCl is the most often-used procedure. Although the commercial treatment removes only a very small percentage of Fe and Al¹⁷, it is definitely beneficial as evidenced by the broad acceptance which the much higher-priced acid-washed supports have found. Less decomposition of solutes (possibly also of the liquid phase) occurs and chromatographic efficiency is generally better.

The detrimental influence of metals can be seen as part chemical reactivity, part surface inhomogeneity. The latter effect appears to be the more difficult to define, but not much is known about either of them. (The great difficulties associated with characterizing inhomogeneity even in pure materials have been pointed out well in recent studies by Waksmundzki *et al.*¹⁸. In our case, several metals on the surface, present in unknown concentrations and chemical states, complicate the matter further.)

We were interested in defining the effects of particular metals present on a diatomaceous support, notably iron and aluminum. To study these effects, a diatomaceous surface covered by only one type of metal, preferably in the oxide or silicate form, had to be available. We were also interested in exploring whether or not our method of "bonding" ultrathin polymer layers on silicic supports could be extended to various types of metal-containing surfaces. (Apparently, it is possible in the case of pure α -Fe₂O₃ (ref. 14).) Finally, we were interested in comparing the chromatographic properties of such surfaces without a modifying polymer layer, *i.e.*, in pure gas-solid chromatography.

A variety of methods can be used to obtain a metal-containing surface. It would be possible, in the case of several metals, to use their oxides in neat form. However, their pore structures may be less than ideal for chromatographic purposes, and phases of different pore structures would be difficult to compare.

It is also possible to coat soluble metal salts onto supports similar to the coating of liquid phases¹. These can be further reacted to yield the desired compounds. Or the desired surface compound can be produced by precipitating it on the support^{8,12}. However, it is apparently very difficult to achieve a homogenous layer with this technique and small, isolated deposits are to be expected⁸.

For our purpose, one promising approach toward the synthesis of fairly "homogenous" layers seemed to be the chemical reaction between surface silanol groups and metal halides in vapor form. This reaction is, in a way, the reverse of our purification technique for GC supports, in which HCl gas reacts at high temperature with the metal oxides and/or silicates and metal chlorides distill off¹⁷.

The reaction of metal halides with surface silanols is not new; such compounds as BCl₃, AlCl₃, etc., have been used for characterizing silanol groups and the water content of silica gels (for a review see ref. 19). Presumably chemical bonding occurred

and an even distribution must have been achieved, reflecting the distribution of silanol groups on the surface.

The skeleton of diatoms consists essentially of silica. Chromosorb, *i.e.*, diatomaceous earth for gas chromatography, should be capable of similar reactions as silica gel. However, Chromosorbs, even in their AW (acid-washed) form, contain significant amounts of Fe, Al and other metals still on their surface. These would render them unusable for the purposes of this study. The clean-up method for Chromosorbs mentioned above removes most of this surface contamination by reaction with hydrogen chloride at 850°¹⁷. The remaining, "clean" silicic surface can be reacted with a particular metal halide and, once a surface layer (possibly with residual, reactive halide) is formed, subsequent reactions can be attempted, *e.g.*, with water to give "oxide", with ammonia to give "nitride" or with hydrogen to give "reduced" type structures. We realized from the beginning that this approach was likely to result in materials whose precise surface structures were impossible to determine by means available to us. Although reactions as cited above are well known in bulk phase, extrapolation to processes occurring in the monomolecular layer region of a silicic substrate, with little-characterized pore geometry and chemical reactivity, is fraught with peril.

Thus, while it is easy to tell that *some* reaction has occurred (say by the evolution of HCl), its exact nature may be uncertain. There may also be the possibilities of incomplete coverage, reaction with the silica network (opening of Si-O-Si), reaction of one average halide molecule with a fractional number (between 0 and 2, generally) of silanol groups, metal valence changes, formation of multiple layers (in a series of reactions alternating metal halide and water, or even with some residual water around) and others more. These ambiguities in mind, we intended this study to serve as a scouting trip into a little-explored field, with chromatography as the main, but not the only, point of interest.

EXPERIMENTAL

Chromosorb W (45-60 mesh in early experiments and 100-120 mesh in later ones) was purified as previously described¹⁷ by a pre-wash with 6 *N* HCl, a treatment with HCl gas at 850°, again a wash with 6 *N* HCl and a final wash with distilled water to neutrality. Then the material was dried overnight at $\approx 150^\circ$ in vacuum.

This Chromosorb (thereafter designated "RW") was filled into a vertically standing quartz tube (about 1 in. diameter, about 1 ft. high) with indentations (to hold the lower quartz wool plug) and ground quartz joint terminals. The upper terminal formed a "T" with the side entrance admitting nitrogen and the main entrance (through a septum) allowing injections, *e.g.*, of water, or permitting a little quartz vessel containing halide to be lowered into the heated region of the quartz tube. The quartz tube was heated by a large aluminum block with cartridge heaters. This oven was supported in a lab-made hood and the exit line from the bottom terminated inside the hood's exhaust duct. It could be easily opened to check for HCl evolution, etc.

The metal halides used in this study were AlCl_3 , BCl_3 , CrO_2Cl_2 , FeCl_3 , MoCl_5 , NbCl_5 , TaCl_5 , SnCl_4 , TiCl_4 , WCl_6 and VOCl_3 . Two non-metal halides processed by the same techniques were POCl_3 and CH_2Cl_2 , the former to produce an acidic phase⁴⁹, the latter to produce a pyrolytic carbon layer²⁰ for purposes of comparison and for

bonding studies. Depending on the volatility of the halide, it was either used as gas or vapor carried by the nitrogen stream or, more often, introduced neat into the top portion of the heated zone where it evaporated. All metal halides were taken through procedures (a) and (b) outlined below; only some of the resulting materials were further processed by (c) or (d).

(a) *Initial reaction with metal halides.* After the tube had been charged with Chromosorb W RW (kept in place by two large quartz wool plugs), nitrogen was turned on to about 30 ml/min for the whole of the experiment. The aluminum block was kept at elevated temperature (the same temperature as used for the subsequent reactions) for 2–6 h, mainly to remove water. Then the little quartz vessel containing the metal halide was lowered into the heated zone (or the nitrogen flow was charged with halide vapor or gas). Hydrogen chloride evolution began and after some time excess metal halide began to appear at the tube exit. This first step was considered complete when the nitrogen stream contained only traces of HCl (usually after 2–4 h). The temperature chosen for this experiment represented a compromise among various considerations involving mainly the volatility and reactivity of the halide and the number and nature of silanol groups on the surface. The resulting material was then treated in one of several ways.

(b) *Further reaction with water.* Keeping the same temperature, an excess of water was injected into the tube (≈ 0.5 ml). Hydrogen chloride evolved and excess water condensed in the exit. It usually took 1–2 h until the HCl content of the exiting N_2 stream was down to trace levels. Then the reaction with metal halide and the subsequent injection of water was repeated. If there was substantially less HCl evolved in the repeated sequence, the procedure was considered complete and the material was left at the elevated temperature overnight and then allowed to cool down. If considerable amounts of HCl were evolved in the second cycle, a third one was added. The nitrogen was kept flowing until the coated material was removed. This type of phase is hereafter called "oxide", although it should be realized that the true structure (which is not known) may not always be fully consistent with this name.

(c) *Further reaction with ammonia.* Starting with material from (a) above, the temperature was raised to the desired level (chosen from ref. 22 for nitride formation from metal halides and NH_3) and approximately 20 ml/min ammonia gas added to the nitrogen stream for about 12 h. Then the ammonia was turned off and the material allowed to cool under nitrogen.

(d) *Further reaction with hydrogen.* This reaction started with the (a) or the (b) type material as described above. In each case, the material was brought to the desired temperature in a suitable quartz tube oven and then ≈ 15 ml/min of hydrogen were added (to the usual 30 ml/min N_2 flow) for 6 h. This type of material is sometimes termed "reduced" in this paper. As in the above cases, the precise state of the surface is unknown.

(e) *Additional heat treatment.* Some phases, especially those that were tested neat rather than be used in polymer bonding experiments, were heated under the usual nitrogen flow to 850° for 12 h in a suitable quartz tube furnace.

Analyses

Due to matrix effects and the extremely small amounts involved, not all materials could be successfully analyzed. In several cases, analyses were performed in

duplicate and on more than one sample. Al, Cr, Fe and Sn were determined by nitrous oxide/acetylene flame emission after extraction with boiling 6 *N* HCl; B, Mo, Nb, P, Ta, Ti, V and W were done by arc spectroscopy. Nitrogen was determined by the Kjeldahl method. These analyses were performed at the University of Missouri Experiment Station's Spectrographic Laboratory (E. E. Pickett) and Chemical Laboratories (C. W. Gehrke). In addition, some analyses of Fe by KMnO₄ titration were done at Dalhousie. A nominal layer thickness was calculated by using the BET surface of the Chromosorb and the bulk density of the compound assumed closest in chemical composition to the layer (*e.g.*, Al₂O₃, CrN, etc.)

Chromatographic tests

All materials were tested as gas chromatographic phases, first neat and then after the attempted synthesis of a bonded Carbowax 20M layer²¹. The chromatographies were done in 1 m × 1.9 mm I.D. borosilicate columns, with 30 ml/min N₂ flow and FID detection in a Shimadzu 4BM gas chromatograph.

RESULTS AND DISCUSSION

As noted in the Experimental section, some elemental analyses were difficult to perform on samples based on Chromosorb W with its low surface area (≈ 1 m²/g). However, in a subsequent investigation involving Porasil A (≈ 400 m²/g) and liquid chromatography²³, more reliable layer thickness values could be obtained. These are comparable to (though, as expected, somewhat lower than) the values obtained on Chromosorb, thus supporting the results of this study.

Following are some values for nominal layer thicknesses with, in parentheses, the colors of the materials, and formulae of the compounds whose densities and element ratios were used for calculation. It should again be noted, though, that the surface layers are not to be equated with these bulk compounds nominally used for computation; the procedure simply reflects reasonable chemical similarities devoid of any experimental corroboration.

Iron was determined in different forms (pink, Fe₂O₃; grey, Fe) and on different samples. All values obtained were in the 1–5 Å range. Chromium gave layer thicknesses of 12 Å (dark reddish brown, CrO₃) and 9 and 10 Å (green, CrN; values for N and Cr, respectively). It should be stressed at this point, however, that we do not know how well the Kjeldahl method was able to cope with this type of sample and that the green color suggests Cr₂O₃. Aluminum showed 6 Å (Al₂O₃). Smaller amounts were found of Ti, Sn, P, V, Mo and B (oxide forms, no obvious color). These most likely reflect layers in the 0.1–1 Å range; with the possible exception of Sn which might have been incompletely extracted, and B, which occurred in the matrix (from the extraction of Chromosorb with 6 *N* HCl in borosilicate soxhlets prior to use). No significant evidence was found for the presence of Nb, Ta and W. Later evidence²³ on silica gel, however, showed Nb and W to be present on that material at 0.04 Å (Nb₂O₅) and about 0.01 Å (brown, WO₂); Ta was not run. Thus, "layers" in the 0.01–0.1 Å range seem likely on Chromosorb. (The layer terminology is kept here for purpose of comparison, even though it has now lost its physical meaning.)

The analyses for Fe, Cr and Al, however, and possibly those for Sn, Ti, V and Mo, also, support the initial assumption that layers in the monomolecular range can be ob-

tained in this fashion. The syntheses performed in this study were run at different temperatures. Temperature influences the number and kind of silanol groups (chemisorbed water), the reaction rate and, because of the quasic chromatographic flow system, the contact time of the metal halide. The reaction conditions were chosen as being "reasonable" but none was optimized. If one assumes a $1 \text{ m}^2/\text{g}$ Chromosorb surface comparable to a fully hydroxylated silica (≈ 6 silanol groups per $100 \text{ \AA}^2$), and calculates the nominal layer thickness of, say, Fe_2O_3 (bulk density $\approx 5 \text{ g/cm}^3$) arising from a load of one metal atom per silanol group, it turns out to be $1.5 \text{ \AA}$. This compares with measured values from $1\text{--}5 \text{ \AA}$. One also needs to realize that not all types of silanol groups are apt to react with all types of metal halides, that certain halide molecules may react with more than one silanol group and that, to start with, the surface is not likely to be fully hydroxylated. All these effects would tend to decrease "layer thickness" *below* its theoretical value. On the other hand, the number of metal atoms bound has been found to be larger in certain cases than the number of silanol groups²⁴. Furthermore, some metal hydroxide structures are undoubtedly formed in the subsequent reaction with water and, again depending on temperature, can react themselves with more metal halide introduced in the second or third cycle. This formation of "multiple layers", and the likely presence of residual adsorbed water as well as the possible presence of residual adsorbed metal halide at different steps in the procedure, all represent effects which would tend to increase "layer thickness" *above* its theoretical value.

It is obvious that a thorough investigation of these parameters would far exceed the confines of a simple chromatographic study. Fortunately, however, some tentative conclusions can be derived from even the scant evidence available.

The major impurities found on the surface of Chromosorbs are iron and aluminum. Both pure iron and aluminum "oxide" could be coated onto pure Chromosorb in relatively thick layers. Throughout the chromatographic tests, the phases based on Fe performed significantly better than the one based on Al; in fact, they were generally comparable to pure Chromosorb itself. Thus the experiments suggest that the presence of Al on the surface of a support is of a considerable greater detriment than the presence of Fe; at least in the context of the tests carried out.

The tests of the metal-containing surfaces themselves, by pure gas-solid chromatography, proved unexciting. Some materials behaved slightly better or just about as well as the pure Chromosorb, others performed considerably worse. Fig. 1 shows a temperature-programmed separation of hydrocarbons on H_2 -treated ("reduced") type layers.

In order to obtain better chromatography, a thin layer of Carbowax 20M can be "bonded" to the surface^{14,21}. We felt that this procedure provided a more interesting test for the various surfaces than the use of standard-load liquid phases. To provide good chromatography it is necessary that Carbowax chains are capable of (a) bonding to the surface and (b) deactivating it. In the case of surfaces with low metal content, "bonding" could occur to the silicic structure (presumably via multipoint hydrogen bonding to the silanol groups), while with high metal content (assuming homogeneous distribution) it would have to involve the surface layer. It is possible, of course, that a particular surface may be capable of good performance with regular (non-bonded) typical GLC liquid phases, but would fail to show such performance in this test because the Carbowax would not bond (= would prove extractable).

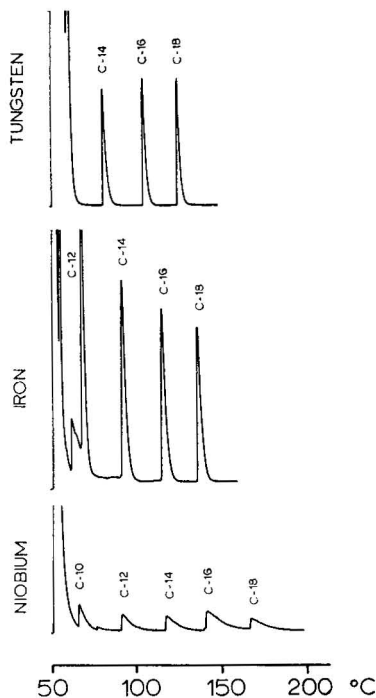


Fig. 1. Temperature-programmed gas chromatographies of *n*-alkanes on Chromosorb W, reacted with metal chlorides and treated with hydrogen as described in the text.

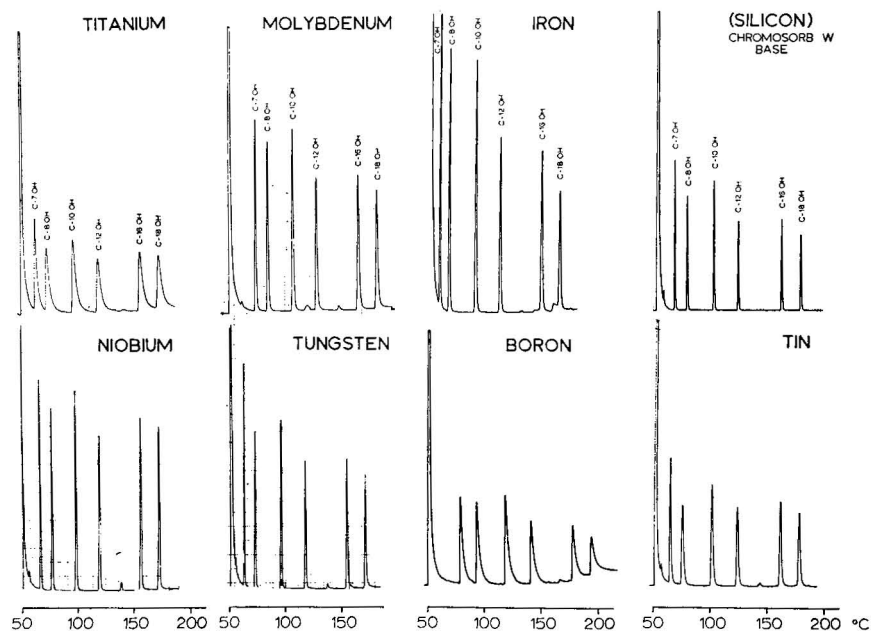


Fig. 2. Temperature-programmed chromatographies of 1-alkanols on Chromosorb W that has been reacted with indicated metal chlorides and water, and has been modified with Carbowax 20M as described in the text.

Fig. 2 shows the temperature-programmed separation of straight-chain primary alcohols on various "oxide" type layers modified by Carbowax 20M and exhaustively extracted with methanol. Some layers, among these the pink, relatively thick one of iron, show acceptable chromatography. Particularly inferior are those based on titanium and boron, and a similar one based on aluminum (not shown) is even worse. This is not completely unexpected in view of the position of these elements in the periodic table and what is known about their surface structures (*e.g.*, ref. 19).

It is not quite clear whether the presence of very small amounts of metals has helped chromatography, but in some cases like tungsten it has at least not hurt it. Fig. 3 shows an isothermal separation of some hydrocarbons and alcohols on the Carbowax-modified surface of Chromosorb W doped with "reduced" tungsten (reaction with WCl_6 , then with H_2). The performance is as good as the best of these types of columns (without metals) can provide; *i.e.*, over 5000 plates per metre, close to the theoretical limit for a 100–120 mesh support. (The flow used is considerably higher than the HETP minimum would suggest; however, the Van Deemter curves for such phases show very low C terms.)

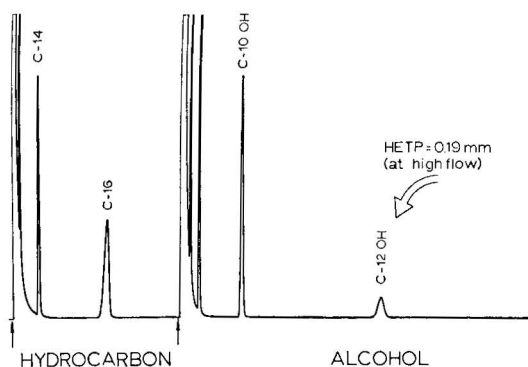


Fig. 3. Comparison of peak shape and efficiency of alkanes and alkanols in isothermal gas chromatography on a 1-m column. Stationary phase: Chromosorb W, 100–120 mesh, doped with WCl_6 , treated with H_2 , and modified with a non-extractable layer from Carbowax 20M.

The performance of a modified, much heavier layer of the "reduced" type, namely the grey layer of iron (reaction with FeCl_3 and H_2O , twice repeated, then treatment with H_2), is shown in Fig. 4 with temperature-programmed separations of two "polarity" test mixtures. It shows that the support is well deactivated and, similar to a bonded layer of Carbowax 20M on pure Chromosorb, considerably less "polar" than a regular Carbowax GLC phase²⁵.

It has been shown before that Carbowax 20M is not the only polymer that can be "bonded" to silicic surfaces²⁶. Even materials as unlikely to bond as Apiezon L can form non-extractable layers on Chromosorb²¹. Now it appears that Carbowax 20M not only bonds to silicic surfaces but also to surfaces which contain various metals in different chemical states. (Even pyrolytic carbon showed a certain affinity for this liquid phase.) Here as there, obviously, particular polymers and particular surfaces are clearly favored over others in terms of chromatographic performance. No attempt has been made to correlate such behavior to, say, the number and reactivity of surface hydroxyls and their steric "fit" to the non-extractable polymer.

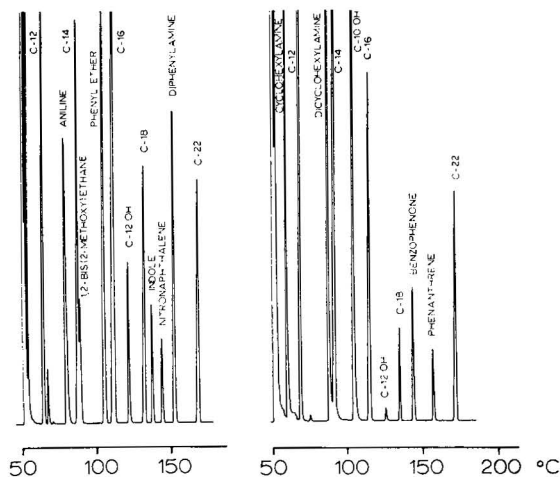


Fig. 4. Temperature-programmed gas chromatographies of two test mixtures containing straight-chain hydrocarbons and a variety of polar compounds. Stationary phase: Chromosorb W reacted with ferric chloride, treated with hydrogen, modified with Carbowax 20M, and packed into a 1 m $\times$ 1.9 mm I.D. borosilicate glass column.

Perhaps some of the metal-containing surfaces may find uses beyond the chromatographic context. They could serve as convenient (and comparable) systems for the study of compound interactions on surfaces by chromatographic means. Then, the chemical reactivity of such surfaces could be interesting to explore—both for catalytic studies and for use as reagents in organic chemistry. (For instance, the colors of a chromium-containing phase once indicated that we had changed, unintentionally, Cr(VI) to Cr(III) on contact with an easily-oxidized GC liquid phase.)

A certain drawback to such studies is undoubtedly the fact that we know little at present about the synthesized surface structures and are unlikely to do so in the near future. The fact that reactions have been obtained is evident from the analysis of the materials, from their chemical and chromatographic behaviour and, in cases like Cr and Fe, from their colour.

It is equally obvious that various other syntheses, similar to the ones used here, could have been easily performed. The choice of what and how to try first was in our case governed by arbitrary factors such as the availability, volatility and easy handling of the particular reagents, our chromatographic interests, hints from the periodic system and known bulk reactions, and the presumed likelihood of success. Many reactions were single attempts because of time constraints, and optimization studies would most likely have improved the product in most cases. While this study was clearly of an exploratory nature, its results seem to justify further interest in the area.

ACKNOWLEDGEMENTS

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NOTE ADDED

Most of this manuscript had been written some time ago and, while literature that has appeared since then does not require any significant change, we would like to add for the convenience of the reader a recent review²⁷ and a few interesting papers²⁸⁻⁴⁸ to the list of references.

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CHROM. 12,419

SILICA GEL MODIFIED WITH PHTHALOCYANINES AS A STATIONARY PHASE IN GAS-SOLID CHROMATOGRAPHY

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SUMMARY

Sorbents have been prepared based on silica gel modified with phthalocyanine and with copper and nickel phthalocyanates. The modification was carried out by adsorbing phthalocyanines from solution and by chemisorbing the *o*-phthalodinitrile on silica gel. Effective sorbents have thus been obtained with high selectivity, especially for separations of nitrogen-containing substances. The modified sorbents have been used in micropacked columns in gas-solid chromatography. *n*-Alkanes, arenes, six-carbon substances of various polarities, alcohols and nitrogen-containing substances were used as sorbates. The advantages of sorbents include a reduction in analysis time, an improvement in peak symmetry, the separation of components of mixtures and the high thermal stability of phthalocyanines.

INTRODUCTION

Silica gel has been used in many chromatographic techniques, as the support for the stationary phase in gas-liquid chromatography or as a sorbent in gas-solid (GSC), thin-layer, high-performance liquid and ion-exchange chromatography, either in its original form or variously modified^{1,2}. The sorption properties of silica gel can be suitably modified by thermal and hydrothermal treatment³⁻⁶ or by chemical modification of its surface, for which a number of substances have been used. The reactivity of the hydroxyl groups in silica gel allows the bonding of a wide variety of organic radicals to the surface, thus giving a large selection of sorbents that are used extensively in both gas and high-performance liquid chromatography⁷⁻¹⁰.

However, sorbents with chemically modified surfaces can also be prepared by employing physical adsorption. Substances with planar molecules are most suitable for this purpose and therefore phthalocyanines (Fig. 1) are also advantageous.

Modification of sorbents with phthalocyanines has been systematically studied by Vidal-Madjar¹¹ and Vidal-Madjar and Guiochon¹², who adsorbed phthalocyanines on a non-specific, non-polar type of sorbent, thermally graphitized carbon black

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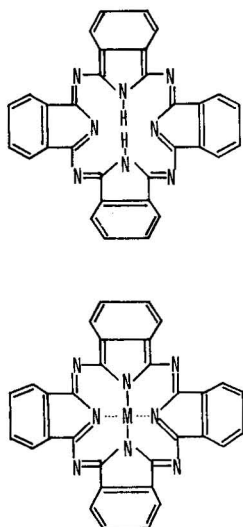


Fig. 1. Molecules of phthalocyanine ($C_{32}N_8H_{18}$) and a metal phthalocyanate ($C_{32}N_8H_{16}M$).

(TGCB). This paper describes the preparation and use of silica gel modified with phthalocyanine and copper and nickel phthalocyanates.

n-Alkanes (pentane to tridecane), arenes (benzene, toluene to butylbenzene), six-carbon ketones and esters and alcohols (methanol to pentanol) were used as sorbates¹³. From the analytical point of view, the separations of nitrogen-containing substances described here appear especially interesting.

EXPERIMENTAL

As the starting material for the preparation of the sorbents, chemically pure Porasil C silica gel (Waters Assoc., Milford, Mass., U.S.A.) was used, with spherical particles 37–75 μ m in diameter and with a uniform porosity, the specific surface area being 75 m²/g. Various procedures were employed for surface modification. Phthalocyanine and copper and nickel phthalocyanates (Research Institute for Organic Syntheses, Pardubice, Czechoslovakia and Bayer, Leverkusen, G.F.R.) were used. The prepared sorbents were packed in glass micropacked columns (295 $\times$ 1 mm I.D.). The sorbates used were API standards.

Chromatographic measurements were performed on a Hewlett-Packard 5700A gas chromatograph equipped with a flame-ionization detector. Nitrogen was used as the carrier gas. Most experiments were carried out at linear flow-rates from 15 to 22 mm/sec, isothermally over a temperature range of 100–250°. The sorbates were injected in the form of saturated vapours at laboratory temperature in amounts of 1–50 μ l using Hamilton syringes.

RESULTS AND DISCUSSION

Methods of modification of silica gel with phthalocyanines

Silica gel was modified with phthalocyanines by adsorption from solution and

by chemisorption of *o*-phthalodinitrile. Phthalocyanines are soluble in only a few inorganic and organic solvents¹⁴. In initial experiments 1-bromonaphthalene was used as the solvent. However, concentrated sulphuric acid was found to be much better as protonation of the nitrogen atoms of the phthalocyanine skeleton makes it possible to prepare sufficiently concentrated phthalocyanine solutions. By reversible deprotonation of the cationic form of the dye produced, the water-insoluble phthalocyanine can again be precipitated. This basic procedure was used in the preparation of the modified sorbents. High purity of all reagents was maintained throughout the preparation.

Sorption from 1-bromonaphthalene solution. For the preparation of a modified sorbent, the solubility of phthalocyanine in 1-bromonaphthalene at an elevated temperature was utilized. The solvent was purified by distillation *in vacuo* at 200° on a 50-cm silica gel column.

Saturated solutions of phthalocyanine in 1-bromonaphthalene were prepared at 250° in a flask fitted with a reflux condenser.

Phthalocyanine was adsorbed on the surface of silica gel by mixing freshly activated silica gel with hot, filtered, saturated solutions of phthalocyanine and copper phthalocyanate in 1-bromonaphthalene. The dye solutions were added in one batch. After 24 h the mixture was filtered and the silica gel was washed with methanol and *n*-heptane and dried *in vacuo* at 150–200°.

Sorption from concentrated sulphuric acid solution. In contrast with the previous procedure, yielding sorbents with a low surface coverage (27 and 41 %), this method produces sorbents with a totally covered surface.

A number of variously modified sorbents was prepared and the surface coverage was determined (see below). From the dependence of the surface coverage on the equilibrium phthalocyanine concentration in sulphuric acid, the optimal concentration of the phthalocyanine solution for the preparation of sorbents with completely modified surfaces was found.

An amount of 0.5 g of phthalocyanine or metal phthalocyanate was dissolved in 5 ml of concentrated sulphuric acid at 70°, under a nitrogen atmosphere and with constant stirring, to prevent oxidation of the phthalocyanine skeleton. Immediately after complete dissolution of the dye, 1 g of silica gel was added to the solution. After cooling of the mixture, the silica gel was isolated and contained the protonated form of phthalocyanine on the surface. Deprotonation was achieved by hydrolysis in a large excess of boiling water (1000 ml) with vigorous stirring. The sedimented modified sorbent was then filtered off on a frit.

In addition to the adsorbed phthalocyanine, the sorbent surface contains a certain amount of mechanically trapped dye, because of the use of concentrated solutions. The excess of dye was separated using the hydrophobicity of phthalocyanines and their insolubility in water, by ultrasonic treatment of the sorbent particles dispersed in water. Excess of dye was removed by a water pump. The sorbent was then dried and activated *in vacuo* at 150–200°.

Phthalocyanine formed from o-phthalodinitrile. The modification of silica gel by chemisorption *o*-phthalodinitrile was also tested.

Silica gel was heated with excess *o*-phthalodinitrile in an electric oven at 390°. The blue-green reaction mixture formed was placed in excess of chloroform (250 ml), in which the dinitrile is readily soluble. The modified silica gel was freed of excess of

unreacted *o*-phthalodinitrile by repeated extraction with chloroform and then dried and activated *in vacuo* at 150–200°.

The dye formed was identified spectrophotometrically in the UV and visible regions (Fig. 2), after extracting the dye formed from 5 g of modified silica gel with sulphuric acid.

The absorption spectrum of the synthesized dye (curve 1) is different from curve 3, corresponding to the initial reactant, confirming that the modified sorbent was sufficiently purified by the extraction from unreacted *o*-phthalodinitrile.

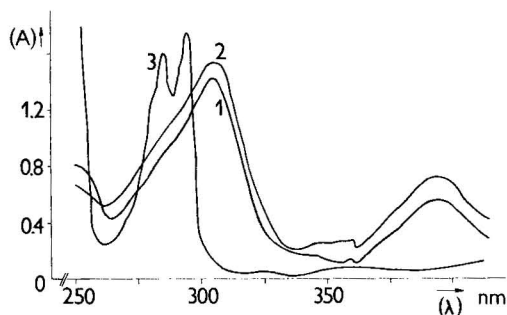


Fig. 2. Absorption spectra of substances dissolved in sulphuric acid: 1 = synthesized dye; 2 = phthalocyanine; 3 = *o*-phthalodinitrile.

However, the agreement of the absorption maxima of curves 1 and 2 is not unambiguous proof of the presence of the phthalocyanine skeleton on the silicagel surface. Therefore, several milligrams of the synthesized dye were isolated by the hydrolytic method and subjected to mass spectrometric analysis. At an ionization energy of 75 eV and an accelerating voltage of 8 kV the existence of a molecular ion with m/e 514, corresponding to phthalocyanine without a central metal atom, was confirmed at 375°.

By chemisorption of *o*-phthalodinitrile, silica gel modified with phthalocyanine was prepared with an average surface coverage of 26%.

Application of the modified sorbents in GSC

The prepared sorbents were used as stationary phases in glass micropacked columns. The surface coverage was found by organic elemental analysis and spectrophotometry, the specific surface area was determined by the thermal desorption method and thus the principal characteristics of the sorbents were obtained (see Table I).

The partially modified sorbents can be used to advantage in analyses of mixtures of alkanes, arenes and various polar substances. However, optimal separations of mixtures of nitrogen-containing substances were achieved only with totally covered sorbents.

Isobutylamine and *n*-pentylamine were separated satisfactorily in a relatively short time at 200 and 250° on Porasil C modified with nickel phthalocyanate (Fig. 3a). The components were not separated on the unmodified sorbent even at 200° and the peak exhibited considerable tailing (Fig. 3b).

TABLE I
PROPERTIES OF THE ADSORBENTS

Adsorbent	Specific surface area (m^2/g)	Surface coverage (%, w/w)*
Phthalocyanine-modified silica gel	123	9.1
Copper phthalocyanate-modified silica gel	111	23.0
Nickel phthalocyanate-modified silica gel	214	40.2
Silica gel	75	—

* The percentages (w/w) of the modifying substance corresponding to the total coverage of the sorbent surface are as follows: for silica gel + $\text{C}_{32}\text{N}_8\text{H}_{18}$, 3.790%; for silica gel + $\text{C}_{32}\text{N}_8\text{H}_{16}\text{Cu}$, 4.207%; and for silica gel + $\text{C}_{32}\text{N}_8\text{H}_{16}\text{Ni}$, 4.213%.

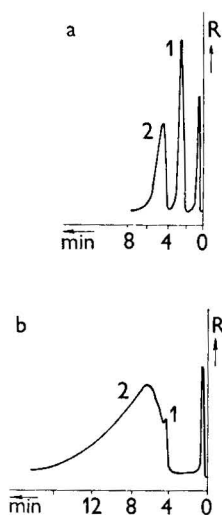


Fig. 3. Separation of a mixture of aliphatic amines: 1 = isobutylamine; 2 = *n*-pentylamine. Micro-packed columns (295×1 mm I.D.); temperature, 200° . (a) Porasil C modified with nickel phthalocyanate, $u = 15.1$ mm/sec; (b) Porasil C, $u = 22.7$ mm/sec.

Aniline and *N,N*-dimethylaniline were partially separated on silica gel at 230° ; their separation improved on decreasing the temperature to 220° (Fig. 4a), but a much better separation was achieved under the same conditions on a column packed with Porasil C modified with free phthalocyanine without a central metal atom (Fig. 4b). The time of analysis can be reduced almost 6-fold by increasing the temperature to 250° , good resolution of the components being retained (Fig. 4c). A similar separation of aromatic amines was obtained on sorbents modified with copper and nickel phthalocyanates; with silica gel modified with copper phthalocyanate the analysis was completed within less than 60 sec (Fig. 5). It should be emphasized that these separations were carried out successfully on micropacked columns less than 30 cm long.

On comparison of the results obtained with the modified silica gel with those obtained on modified TGCB, it can be seen that in both instances the modification caused a change in the sorbate-sorbent interaction and hence also a change in the sorption properties of the materials. Non-specific TGCB was converted into a sorbent



Fig. 4. Separation of a mixture of aromatic amines: 1 = aniline; 2 = N,N-dimethylaniline. Micro-packed columns (295×1 mm I.D.). (a) Porasil C, 220° , $u = 20.3$ mm/sec; (b) Porasil C modified with phthalocyanine, 220° ; (c) Porasil C modified with phthalocyanine, 250° .

Fig. 5. Separation of a mixture of aromatic amines: 1 = aniline; 2 = N,N-dimethylaniline. Micro-packed columns (295×1 mm I.D.); temperature, 200° . Porasil C modified with copper phthalocyanate, $u = 73.8$ mm/sec.

that allowed specific interactions, resulting in a change in the order of elution of the components of the mixture¹¹.

Silica gel is a specific sorbent with a much more heterogeneous surface than TGCB. However, good agreement was attained in separations of arenes and nitrogen-containing substances on the two sorbents. Modification of the silica gel with phthalocyanines led to especially good results in analyses of mixtures of nitrogen-containing substances (see Figs. 3–5). It also follows from Fig. 3a and b that the modification produced a totally covered sorbent surface, eliminating the negative effect of the hydroxyl groups, which is manifested in non-symmetry of polar sorbate peaks.

The positive effect of specific interactions of phthalocyanines was especially marked in the analysis of a mixture of aniline and N,N-dimethylaniline, where the two components were completely separated owing to the specificity of the modified sorbent (Fig. 4a and b).

Because of the use of micropacked columns, the time of analysis was substantially shortened compared with the work of Vidal-Madjar¹¹. The separation of aromatic amines took less than 60 sec, retaining good separation efficiency (see Fig. 5), i.e., the analysis time was shortened 20-fold with only a 4-fold increase in the linear flow-rate.

Vetrova *et al.*¹⁵ also used silica gel (Silochrom-80) modified with phthalocyanine and metal phthalocyanates and obtained the best results with a sorbent modified with copper chlorophthalocyanate. However, this material is thermally unstable and can be used only up to 130° , whereas our materials yield reproducible results even at temperatures up to 250° . Vetrova *et al.* found that polar substances

are eluted from sorbents modified with copper and zinc phthalocyanates with non-symmetrical peaks and long retention times. These negative effects were attributed to impurities in the initial material. We consider that the good results obtained in the present work are due not only to the use of Porasil C, which is a chemically pure material, but also because sorbents with a totally covered surface were prepared, thus eliminating the negative effect of the hydroxyl groups.

Further possibilities of using phthalocyanines

Planar phthalocyanine molecules could be used for separation of geometric isomers, could widen the range of mobile phases used in high-performance liquid chromatography and could probably find some use in ion-exchange chromatography. These possibilities and the interpretation of the sorbate-sorbent interaction mechanism are being studied.

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CHROM. 12,443

LIQUID ION EXCHANGERS IN PAPER CHROMATOGRAPHY OF STEROIDAL GLUCOSIDURONIC ESTERS

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SUMMARY

Twenty-five steroidal glucosiduronic esters were chromatographed on paper in each of five straight-phase systems and five reversed-phase systems. The resolving characteristics of these systems were compared by expressing the retention values in terms of R_M and correlating the data by use of linear regression equations. The resolving characteristics of the straight-phase systems as a group are very similar, and those of the reversed-phase systems are somewhat less similar; the various systems differ considerably in polarity. The resolving properties of a straight-phase system and a reversed-phase system which contain the same liquid ion exchanger are markedly different, a circumstance which increases the probability that a pair of compounds which was not separated in a straight-phase system will be resolved in the reversed-phase system which contains the same liquid ion exchanger. The migration of the esters in the foregoing systems was compared with the migration of the corresponding glucosiduronic acids (a previous study) in the same solvent systems. If a particular pair of conjugates is not separable as carboxylic acids in a specific straight-phase system, there is a relatively good chance that they will be separable as methyl esters in the same system.

INTRODUCTION

In previous studies we chromatographed a collection of steroidal glucosiduronic acids in a group of straight-phase^{1–3} and reversed-phase⁴ solvent systems and compared the resolving properties of the various systems. More recently, we have considered whether a mixture of these steroidal glucosiduronates would be more readily resolved in the form of methyl esters than as the carboxylic acids. Consequently, we chromatographed the methyl esters in each of five straight-phase systems and five reversed-phase systems and compared the extent of resolution achieved on a mixture of conjugates in the form of free acids with that achieved in the form of methyl esters. This paper gives a resumé of our findings. The resolving properties of the various solvent systems are correlated by use of linear regression equations.

MATERIALS AND METHODS

Sources of ion exchangers, glucosiduronic esters, and other chemicals have been given previously¹⁻³. Chromatographic procedures which were described earlier for straight-phase¹⁻³ and for reversed-phase⁴ systems were followed. The steroidal glucosiduronic esters which were chromatographed are listed in Table I. Straight-phase systems, which consisted predominantly of chloroform and formamide, were modified by making the mobile phase of 0.10 *N* with an ion exchanger*; no counterion was added to the formamide phase. Reversed-phase systems employed 0.20 *M* aqueous KCl as mobile phase on sheets of paper which had been dipped in a 0.050 *M* solution of exchanger** in chloroform and allowed to dry in air for about 5 min before the compounds to be chromatographed were applied.

TABLE I

LIST OF STEROIDAL GLUCOSIDURONIC ESTERS

The numbers used for the esters correspond to those assigned to the acids in previous papers^{1,4}.

Ester Name

1	Methyl (3,20-dioxopregn-4-en-21-yl β -D-glucopyranosid)uronate
2	Methyl (17-hydroxy-3,20-dioxopregn-4-en-21-yl β -D-glucopyranosid)uronate
3	Methyl (21-hydroxy-20-oxo-5 β -pregnan-3 α -yl β -D-glucopyranosid)uronate
4	Methyl (3 α -hydroxy-20-oxo-5 β -pregnan-21-yl β -D-glucopyranosid)uronate
5	Methyl (3,11,20-trioxopregn-4-en-21-yl β -D-glucopyranosid)uronate
6	Methyl (11 β -hydroxy-3,20-dioxopregn-4-en-21-yl β -D-glucopyranosid)uronate
7	Methyl (17-hydroxy-3,11,20-trioxopregn-4-en-21-yl β -D-glucopyranosid)uronate
8	Methyl (3 α -17-dihydroxy-20-oxo-5 β -pregnan-21-yl β -D-glucopyranosid)uronate
9	Methyl [(18R)(11 β ,18-epoxy-21-hydroxy-3,20-dioxopregn-4-en-18-yl α -D-glucopyranosid)]-uronate
10	Methyl (3 α -hydroxy-11,20-dioxo-5 β -pregnan-21-yl β -D-glucopyranosid)uronate
11	Methyl (21-hydroxy-11,20-dioxo-5 β -pregnan-3 α -yl β -D-glucopyranosid)uronate
12	Methyl (11 β ,17-dihydroxy-3,20-dioxopregn-4-en-21-yl β -D-glucopyranosid)uronate
13	Methyl (17,21-dihydroxy-20-oxo-5 β -pregnan-3 α -yl β -D-glucopyranosid)uronate
14	Methyl (3 α ,11 β -dihydroxy-20-oxo-5 β -pregnan-21-yl β -D-glucopyranosid)uronate
15	Methyl (3 α ,17-dihydroxy-11,20-dioxo-5 β -pregnan-21-yl β -D-glucopyranosid)uronate
16	Methyl (17,21-dihydroxy-11,20-dioxo-5 β -pregnan-3 α -yl β -D-glucopyranosid)uronate
17	Methyl (20 β ,21-dihydroxy-11-oxo-5 β -pregnan-3 β -yl β -D-glucopyranosid)uronate
18	Methyl (11 β ,21-dihydroxy-20-oxo-5 β -pregnan-3 α -yl β -D-glucopyranosid)uronate
20	Methyl (3 α ,11 β ,17-trihydroxy-20-oxo-5 β -pregnan-21-yl β -D-glucopyranosid)uronate
21	Methyl (17,20 β ,21-trihydroxy-11-oxo-5 β -pregnan-3 α -yl β -D-glucopyranosid)uronate
22	Methyl (11 β ,17,21-trihydroxy-20-oxo-5 β -pregnan-3 α -yl β -D-glucopyranosid)uronate
24	Methyl (11 β ,17,20 β ,21-tetrahydroxy-5 β -pregnan-3 α -yl β -D-glucopyranosid)uronate
26	Dimethyl (20-oxo-5 β -pregnan-3 α ,21-ylene di- β -D-glucopyranosid)uronate
27	Dimethyl (11,20-dioxo-5 β -pregnan-3 α ,21-ylene di- β -D-glucopyranosid)uronate
28	Dimethyl (11 β -hydroxy-20-oxo-5 β -pregnan-3 α ,21-ylene di- β -D-glucopyranosid)uronate

* Straight-phase systems are designated by the exchanger employed and the subscript SP. Exchangers used in straight-phase systems were: tetraheptylammonium acetate (TA·OAc); tetraheptylammonium chloride (TA·Cl); tetraheptylammonium sulfate [(TA)₂·SO₄]; tri-*n*-octylamine hydrochloride (TOA·HCl); and tri-*n*-octylamine hydrosulfate [(TOA·H)₂SO₄].

** Reversed-phase systems are designated by the exchanger employed and the subscript RP. Exchangers used for reversed-phase systems were: Amberlite LA-2 hydrochloride (ALA-2·HCl); Amberlite XLA-3 hydrochloride (XLA-3·HCl); methyltricaprylammonium chloride (Aliquat); TA·Cl; and TOA·HCl.

RESULTS AND DISCUSSION

R_F values were determined for 22 monoglucosiduronic esters and three diglucosiduronic esters in five straight-phase systems (Table II) and in five reversed-phase

TABLE II

R_F AND R_M VALUES OF STEROIDAL GLUCOSIDURONIC ESTERS IN STRAIGHT-PHASE SYSTEMS

The mobile phase was 0.10 *N* ion exchanger in chloroform; stationary phase was formamide. Glucosiduronic esters are listed in Table I.

Ester System

	$TOA \cdot HCl_{SP}$		$(TOA \cdot H)_2SO_{4SP}$		$TA \cdot Cl_{SP}$		$TA \cdot OAc_{SP}$		$(TA)_2 \cdot SO_{4SP}$	
	R_F	R_M	R_F	R_M	R_F	R_M	R_F	R_M	R_F	R_M
1	0.38	0.21	0.63	-0.23	0.77	-0.53	0.84	—	—	—
2	0.067	1.14	0.23	0.53	0.32	0.33	0.68	-0.33	0.74	-0.45
3	0.33	0.31	0.57	-0.12	0.72	-0.41	0.81	—	—	—
4	0.14	0.79	0.35	0.27	0.49	0.02	0.73	-0.43	—	—
5	0.13	0.83	0.34	0.29	0.45	0.09	0.74	-0.45	—	—
6	0.055	1.24	0.20	0.60	0.30	0.37	0.69	-0.35	—	—
7	0.026	1.57	0.11	0.91	0.18	0.66	0.55	-0.09	0.64	-0.25
8	0.020	1.69	0.069	1.13	0.12	0.87	0.44	0.10	0.57	-0.12
9	0.090	1.00	0.33	0.31	0.35	0.27	0.71	-0.39	—	—
10	0.032	1.48	0.11	0.91	0.19	0.63	0.53	-0.05	0.64	-0.25
11	0.072	1.11	0.23	0.53	0.28	0.41	0.63	-0.23	0.69	-0.35
12	0.010	—	0.038	1.40	0.068	1.14	0.33	0.31	0.54	-0.07
13	0.034	1.45	0.14	0.79	0.21	0.58	0.55	-0.09	0.67	-0.31
14	0.014	—	0.053	1.25	0.080	1.06	0.36	0.25	0.51	-0.02
15	—	—	0.026	1.57	0.052	1.26	0.27	0.43	0.36	0.25
16	0.011	—	0.046	1.32	0.086	1.03	0.39	0.19	0.53	-0.05
17	—	—	0.053	1.25	0.10	0.95	0.45	0.09	0.53	-0.05
18	0.026	1.57	0.090	1.00	0.12	0.87	0.43	0.12	0.52	-0.04
20	—	—	—	—	0.016	1.79	0.12	0.87	0.25	0.48
21	—	—	0.018	1.74	0.030	1.51	0.26	0.45	0.41	0.16
22	—	—	0.013	—	0.021	1.67	0.13	0.83	0.26	0.45
24	—	—	—	—	—	—	0.10	0.95	0.27	0.43
26	—	—	0.044	1.34	0.059	1.20	0.51	-0.02	0.65	-0.27
27	—	—	0.014	—	0.026	1.57	0.31	0.35	0.54	-0.07
28	—	—	—	—	—	—	0.14	0.79	0.36	0.25

systems (Table III). In these tables mobility of the compounds is expressed also in terms⁵ of R_M ; $R_M = \log [(1/R_F) - 1]$. The properties of two chromatography systems relative to a series of compounds can be compared by plotting values for migration in one system against those in another system. If mobility is expressed as R_F , a curvilinear relationship is usually obtained; when mobility is expressed as R_M , a linear relationship is commonly observed. Rather than plotting the R_M values on a graph, we have elected to use the R_M values in linear regression equations for comparing the chromatographic characteristics of the various solvent systems with those of one solvent system which is taken as a standard. The equations, which are analogous to those

TABLE III

R_F AND R_M VALUES OF STEROIDAL GLUCOSIDURONIC ESTERS IN REVERSED-PHASE SYSTEMS

The mobile phase was 0.20 *M* aqueous KCl; the paper was impregnated with 0.050 *M* exchanger in chloroform. Glucosiduronic esters are listed in Table I.

Ester	$TA \cdot Cl_{RP}$		$Aliquat_{RP}$		$TOA \cdot HCl_{RP}$		$ALA-2 \cdot HCl_{RP}$		$XLA-3 \cdot HCl_{RP}$	
	R_F	R_M	R_F	R_M	R_F	R_M	R_F	R_M	R_F	R_M
1	0.028	1.54	0.048	1.30	0.20	0.60	0.48	0.04	0.45	0.09
2	0.019	1.71	0.057	1.22	0.18	0.66	0.50	0.00	0.58	-0.14
3	—	—	0.013	—	0.027	1.56	0.12	0.87	0.16	0.72
4	—	—	0.011	—	0.024	1.61	0.12	0.87	0.12	0.87
5	0.15	0.75	0.25	0.48	0.55	-0.09	0.72	-0.41	0.69	-0.35
6	0.048	1.30	0.078	1.07	0.31	0.35	0.61	-0.19	0.59	-0.16
7	0.077	1.08	0.23	0.53	0.50	0.00	0.72	-0.41	0.73	-0.43
8	—	—	0.014	—	0.032	1.48	0.18	0.66	0.23	0.53
9	0.26	0.45	0.32	0.33	0.63	-0.23	0.75	-0.48	0.71	-0.39
10	0.029	1.53	0.049	1.29	0.22	0.55	0.49	0.02	0.43	0.12
11	0.041	1.37	0.061	1.19	0.27	0.43	0.56	-0.10	0.50	0.00
12	0.035	1.44	0.10	0.95	0.30	0.37	0.64	-0.25	0.67	-0.31
13	—	—	0.014	—	0.030	1.51	0.16	0.72	0.26	0.45
14	0.019	1.71	0.040	1.38	0.16	0.72	0.49	0.02	0.46	0.07
15	0.024	1.61	0.075	1.09	0.21	0.58	0.59	-0.16	0.59	-0.16
16	0.028	1.54	0.070	1.12	0.23	0.53	0.59	-0.16	0.61	-0.19
17	0.031	1.50	0.057	1.22	0.25	0.48	0.59	-0.16	0.49	0.02
18	0.018	1.74	0.047	1.31	0.16	0.72	0.52	-0.04	0.53	-0.05
20	0.024	1.61	0.069	1.13	0.18	0.66	0.58	-0.14	0.58	-0.14
21	0.058	1.21	0.13	0.83	0.44	0.10	0.72	-0.41	0.66	-0.29
22	0.027	1.56	0.081	1.06	0.23	0.53	0.62	-0.21	0.64	-0.25
24	0.072	1.11	0.15	0.75	0.46	0.07	0.74	-0.45	0.69	-0.35
26	—	—	0.018	1.74	0.12	0.87	0.49	0.02	0.27	0.43
27	0.074	1.10	0.11	0.91	0.54	-0.07	0.76	-0.50	0.58	-0.14
28	0.040	1.38	0.088	1.02	0.45	0.09	0.75	-0.48	0.58	-0.14

discussed in detail previously¹, are of the following general form where symbols *a* and *b* represent slope and intercept in the equation for a straight line.

$$R_M(\text{system } Y) = a \cdot R_M(\text{system } X) + b \quad (1)$$

Of primary importance in the interpretation of regression equations is the coefficient of correlation, *r*, which may range from 1.00 (perfect correlation) to 0.00 (no correlation) to -1.00 (perfect inverse correlation). In the present application, the commonly used coefficient *r* is a curvilinear measure of the degree of similarity in the chromatographic migration of a group of compounds in two solvent systems (*X* and *Y*). The expression $\sqrt{1-r^2}$ is a linear measure of the degree of similarity in the resolving properties of two systems, and thus it is simpler to interpret. When applied to chromatographic data for a group of compounds in two systems, $\sqrt{1-r^2}$ gives the fractional amount of the resolving power⁶ of system *Y*, in terms of standard deviation of mean

R_M , which is unique and useful for separating a mixture of components not separated by system X . When applied to chromatographic data from a single system on two related groups of compounds (e.g., parent compounds and homogeneous derivatives), $\sqrt{1-r^2}$ gives the fractional amount of the resolving power of the system which is unique for the derivatives and is useful for separating as derivatives mixtures that were not separated as parent compounds*.

Fig. 1 shows the relationship between values for r and $\sqrt{1-r^2}$. It is apparent that as values for r decrease from 1.00 to slightly lower amounts, corresponding values for $\sqrt{1-r^2}$ increase very rapidly. Thus, when $r = 0.97$, $\sqrt{1-r^2} \approx 0.25$, and when $r = 0.85$, $\sqrt{1-r^2} \approx 0.50$. A value of 0.50 for $\sqrt{1-r^2}$ implies that, in terms of R_M , 50% of the intrinsic resolving power of system Y is available for resolving a mixture of compounds not separated by system X .

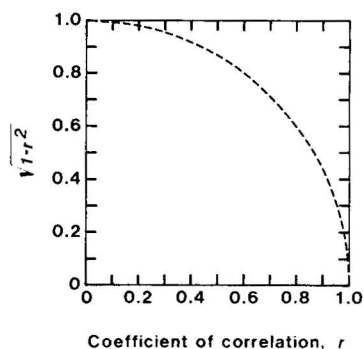


Fig. 1. Relationship between r and $\sqrt{1-r^2}$ for application to linear regression analysis.

An additional attribute which may be useful in describing eqn. 1 is a/r ; this value expresses the ratio of the standard deviation of the mean R_M in system Y to the standard deviation of the mean R_M in system X . If the value for a/r is larger than 1.00, system Y offers the greater probability in terms of R_M of separating two randomly selected substances; if the value is less than 1.00, system X is superior.

The data on the monoglucosiduronic esters (compounds 1-24) in Table II have been evaluated by designating the $\text{TA} \cdot \text{Cl}_{\text{SP}}$ systems as system X in eqn. 1 and deriving regression equations for the four other systems relative to this standard. The resulting equations are summarized in Table IV. In each case, r is greater than 0.97, a value which indicates that the resolving properties of the five systems are very similar (see Fig. 1). Values for a/r indicate that the $\text{TOA} \cdot \text{HCl}_{\text{SP}}$ and $(\text{TOA} \cdot \text{H})_2\text{SO}_{4\text{SP}}$ systems give slightly better resolution of the esters than the standard system and that the $\text{TA} \cdot \text{OAc}_{\text{SP}}$ and $(\text{TA})_2 \cdot \text{SO}_{4\text{SP}}$ systems give poorer resolution. Examination of the values for R_F in Table II, or values for b in Table IV, show that the systems differ markedly in polarity, $\text{TOA} \cdot \text{HCl}_{\text{SP}}$ being least polar and $(\text{TA})_2 \cdot \text{SO}_{4\text{SP}}$ being most polar.

* Simultaneously, $\sqrt{1-r^2}$ gives the fractional amount of resolving power which is unique for parent compounds and is useful for separating as parent compounds mixtures which were not separated as derivatives.

TABLE IV

COMPARISON OF STRAIGHT-PHASE SYSTEMS FOR CHROMATOGRAPHY OF STEROIDAL MONOGLUCOSIDURONIC ESTERS

Values in the table apply to the equation $R_M(\text{system } Y) = a \cdot R_M(\text{TA} \cdot \text{Cl}_{\text{SP}}) + b$; r is the coefficient of correlation; n is the number of compounds considered in the correlation.

System Y	a	b	r	n
TOA $\cdot$ HCl _{SP}	1.07	0.76	0.990	13
(TOA $\cdot$ H) ₂ SO _{4SP}	1.01	0.22	0.992	19
TA $\cdot$ OAc _{SP}	0.76	-0.56	0.987	19
(TA) ₂ $\cdot$ SO _{4SP}	0.61	-0.64	0.971	15

For correlation of the data on the reversed-phase systems (Table III), TA $\cdot$ Cl_{RP} was used as system X in eqn. 1; the resulting regression equations are summarized in Table V. Values for r indicate that systems designated ALA-2 $\cdot$ HCl_{RP} and XLA-3 $\cdot$ HCl_{RP} have resolving properties for the esters that are considerably different from those of the standard system. The best resolution of the esters by a reversed-phase

TABLE V

COMPARISON OF REVERSED-PHASE SYSTEMS FOR CHROMATOGRAPHY OF STEROIDAL GLUCOSIDURONIC ESTERS

Values in the table apply to the equation $R_M(\text{system } Y) = a \cdot R_M(\text{TA} \cdot \text{Cl}_{\text{RP}}) + b$; r is the coefficient of correlation; n is the number of compounds considered in the correlation.

System Y	a	b	r	n
Aliquat _{RP}	0.83	-0.13	0.927	18
TOA $\cdot$ HCl _{RP}	0.82	-0.74	0.965	18
ALA-2 $\cdot$ HCl _{RP}	0.42	-0.77	0.835	18
XLA-3 $\cdot$ HCl _{RP}	0.34	-0.63	0.673	18

system, as indicated by values for a/r , is achieved by use of TA $\cdot$ Cl_{RP}. The general order of decreasing polarity (*i.e.*, increasing R_F for reversed-phase systems) is TA $\cdot$ Cl_{RP} > Aliquat_{RP} > TOA $\cdot$ HCl_{RP} > ALA-2 $\cdot$ HCl_{RP} $\approx$ XLA-3 $\cdot$ HCl_{RP}.

$$R_M(\text{TOA} \cdot \text{HCl}_{\text{RP}}) = -0.095 R_M(\text{TOA} \cdot \text{HCl}_{\text{SP}}) + 0.81; n = 13; r = -0.069 \quad (2)$$

$$R_M(\text{TA} \cdot \text{Cl}_{\text{RP}}) = 0.20 R_M(\text{TA} \cdot \text{Cl}_{\text{SP}}) + 1.23; n = 17; r = 0.349 \quad (3)$$

Comparison of the mobility of the esters in a straight-phase system with their mobility in a reversed-phase system contains the same exchanger is shown in eqns. 2 and 3. It is apparent from the values for r that there is very little correlation between the migration of the esters in corresponding straight-phase and reversed-phase systems. This observation is supported by comparison of data⁷ from the corresponding pairs of systems which employ Aliquat, ALA-2 $\cdot$ HCl and XLA-3 $\cdot$ HCl in which $r = 0.054$, -0.45 and -0.42 , respectively.

Linear regression equations of the following general form were used also to compare the chromatographic migration of the monoglucosiduronic esters with that

of the corresponding monoglucosiduronic acids in the same solvent system or in comparable* systems.

$$R_M(\text{Ester}) = a \cdot R_M(\text{Acid}) + b \quad (4)$$

The equations are summarized in Table VI. For all of the straight-phase systems and the Aliquat_{RP} system, values of r indicate a moderate correlation between migration of the glucosiduronates as esters and as acids ($0.74 < r < 0.89$). Interpretation of these values of r in terms of $\sqrt{1 - r^2}$ (see Fig. 1) implies that if an arbitrarily chosen pair of conjugates was not separated in a particular solvent system as glucosiduronic acids that about half of the intrinsic resolving power of the system would be available to separate the pair as glucosiduronic esters.

TABLE VI

COMPARISON OF THE MIGRATION OF GLUCOSIDURONIC ACIDS AND ESTERS IN COMPARABLE SYSTEMS

Values are from the equation $R_M(\text{ester}) = a \cdot R_M(\text{acid}) + b$. Data are from Tables II and III and refs. 1 and 4.

System	a	b	r	a/r	n
<i>A. Straight-phase</i>					
TOA·HCl _{SP}	0.81	0.57	0.854	0.95	13
(TOA·H) ₂ SO _{4SP}	0.84	0.57	0.869	0.97	18
TA·Cl _{SP}	1.03	-0.03	0.884	1.17	21
TA·OAc _{SP}	0.85	0.10	0.859	0.99	19
(TA) ₂ ·SO _{4SP}	0.86	0.30	0.749	1.15	13
<i>B. Reversed-phase</i>					
TA·Cl _{RP}	0.88	0.51	0.967	0.91	18
Aliquat _{RP}	0.68	0.35	0.859	0.79	18
TOA·HCl _{RP}	0.92	-0.15	0.973	0.95	22
ALA-2·HCl _{RP}	0.79	-0.30	0.981	0.81	22
XLA-3·HCl _{RP}	0.65	-0.54	0.954	0.68	22

In the straight-phase systems (Table VI), values for a/r indicate that the conjugates are resolved as well in the form of esters as in the form of acids. However, in the reversed-phase systems, the conjugates are resolved better as acids.

It has been postulated⁸⁻¹⁰ that if there were no interaction among the various structural components of a molecule, the change in R_M (ΔR_M) associated with a structural alteration at a single location would be constant. Under such circumstances, ΔR_M of esterification for a group of glucosiduronic acids in a specific solvent system should be a constant. The equation relating migration of the esters to that of the acids in a specific system would be $R_M(\text{ester}) = 1.00 R_M(\text{acid}) + \Delta R_M$ and the coefficient of correlation would be 1.00.

* The straight-phase systems used for the acids¹ consisted of 0.10 *N* exchanger in chloroform and 0.10 *N* counterion in formamide. Since addition of counterion to the stationary phase does not affect the migration of the esters significantly³, the counterion was omitted from the systems used with the esters.

A summary of ΔR_M values obtained from chromatography of the glucosiduronic acids and esters in four solvent systems is given in Table VII. When all of the conjugates are included in one group (Table VII, line 1), there is a large average deviation from the mean ΔR_M value with three of the systems and a small deviation with the other one. If the glucosiduronates are grouped according to various structural similarities of the compounds, the average deviations of the mean ΔR_M values are reduced with each of the four systems; the values are reduced markedly with the three systems which initially had large average deviations. This observation is illustrated graphically for the $TA \cdot Cl_{SP}$ system in Fig. 2. In the regression equation for the line in the left panel (all conjugates in one group), the value of r is 0.884; values of r for lines in the right panel (conjugates grouped by structural similarities) are greater than 0.990. Analogous relationships between constancy of ΔR_M and structure are observed when the data from the other straight-phase systems, or from the $Aliquat_{RP}$ system are plotted in a similar manner (plots not shown).

TABLE VII

MEAN AND AVERAGE DEVIATION OF ΔR_M VALUES FOR CONVERSION OF GLUCOSIDURONIC ACID TO GLUCOSIDURONIC METHYL ESTER

Data are from Tables II and II and from refs. 1 and 4.

Types of compounds compared	System			
	$TA \cdot Cl_{SP}$	$(TOA \cdot H)_2SO_{ASP}$	$Aliquat_{RP}$	$TA \cdot Cl_{RP}$
All monoglucosiduronates	0.01 ± 0.25	-0.52 ± 0.23	-0.04 ± 0.18	-0.40 ± 0.08
17-Hydroxy compounds conjugated at C-21	-0.37 ± 0.03	-0.84 ± 0.04	0.15 ± 0.03	-0.32 ± 0.03
11 β -Hydroxy-17-deoxy compounds conjugated at C-21	-0.17 ± 0.03	-0.74 ± 0.02	0.08 ± 0.01	-0.30 ± 0.03
17-Deoxy compounds conjugated at C-21 with either 11-deoxy or 11-oxo group	0.14 ± 0.04	-0.51 ± 0.01	0.05 ± 0.06	-0.34 ± 0.02
17-Hydroxy compounds conjugated at C-3	0.10 ± 0.06	-0.28 ± 0.03	-0.23 ± 0.06	-0.47 ± 0.07
17-Deoxy compounds conjugated at C-3	0.36 ± 0.09	-0.25 ± 0.03	-0.19 ± 0.05	-0.49 ± 0.05

By using the data in ref. 1 and in Table II, values were calculated for the ΔR_M of hydroxylation at C-17 in the glucosiduronic acids and esters in the $TA \cdot Cl_{SP}$ system. The mean values for ΔR_M of hydroxylation at C-17 in six pairs of C-21 conjugates and in four pairs of C-3 conjugates are given below.

$$\begin{aligned}\Delta R_M (17\alpha\text{-hydroxylation}) \text{ for esters conjugated at C-21} &= 0.74 \\ \Delta R_M (17\alpha\text{-hydroxylation}) \text{ for acids conjugated at C-21} &= 0.32 \\ \text{Difference} &= 0.42\end{aligned}$$

$$\begin{aligned}\Delta R_M (17\alpha\text{-hydroxylation}) \text{ for esters conjugated at C-3} &= 0.74 \\ \Delta R_M (17\alpha\text{-hydroxylation}) \text{ for acids conjugated at C-3} &= 0.49 \\ \text{Difference} &= 0.25\end{aligned}$$

These values indicate that in the esters a 17 α -hydroxyl group is more polar (retards migration more) than in the acids. In addition, the values also indicate that while position of conjugation has no appreciable effect on polarity of the 17 α -hydroxyl group in the esters, the function is less polar in acids conjugated at C-21 than in acids conjugated at C-3. The foregoing comparisons provide an example of strong interactions among the various structural elements within a compound being chromato-

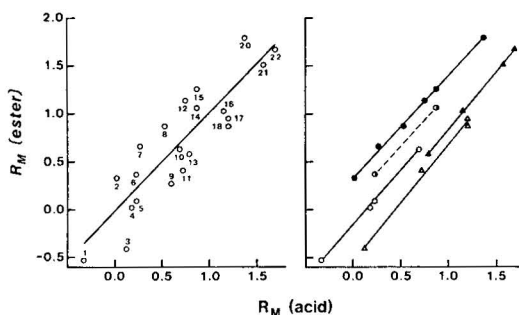


Fig. 2. Comparison of the chromatographic migration (R_M scale) of steroidal glucosiduronic esters and glucosiduronic acids in comparable straight-phase systems containing TA·Cl; data are from Table II and ref. 1. In the left panel, the conjugates are compared as a group; the equation of the line is $R_M(\text{ester}) = 1.03 R_M(\text{acid}) - 0.02$. In the right panel, the conjugates are divided according to the following structural similarities: ●, 17-hydroxy compounds conjugated at C-21; ◐, 11 β -hydroxy-17-deoxy compounds conjugated at C-21; ○, 17-deoxy compounds conjugated at C-21 with either 11-deoxy or 11-oxo group; ▲, 17-hydroxy compounds conjugated at C-3; △, 17-deoxy compounds conjugated at C-3. The equations of the lines are: ●, $R_M(\text{ester}) = 1.07 R_M(\text{acid}) + 0.33$; ○, $R_M(\text{ester}) + 1.14 R_M(\text{acid}) - 0.16$; ▲, $R_M(\text{ester}) + 1.20 R_M(\text{acid}) - 0.36$; △, $R_M(\text{ester}) = 1.21 R_M(\text{acid}) - 0.53$.

graphed and/or of large differences in the extent to which some of the structural elements interact with components of the solvent system. The variability of the interactions in glucosiduronic acids which are conjugated at different positions, as opposed to findings in the esters, leads to a variability in ΔR_M values and makes accurate prediction of R_M values for the compounds impossible. However, these same variations of interaction make it possible, in some cases, to separate a pair of substances after they have been derivatized when it was not possible to separate them as the parent compounds.

ACKNOWLEDGEMENTS

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BESTIMMUNG DER KURZKETTIGEN MONOCARBONSÄUREN AUS NATÜRLICH VORKOMMENDEN ESTERN DURCH GAS-LIQUID-SOLID-CHROMATOGRAPHIE AN MIKROGEPACKTEN SÄULEN

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SUMMARY

Determination of free short-chain monocarboxylic acids from naturally occurring esters by gas-liquid-solid chromatography on micropacked columns

A new gas-liquid-solid chromatographic method (stationary phase 6% OV-210 on Chromosorb 101) allows the separation and determination of acetic, propionic, isobutyric, *n*-butyric, 2-methylbutyric, isovaleric, *n*-valeric, isocaproic, *n*-caproic, 3,3-dimethylacrylic, angelic and tiglic acid by using micropacked glass columns without mixing carrier gas with formic acid vapour. Quantification is obtained by means of internal standard, the lower limit of linear range being 0.1 µg acid per injection.

The general applicability of this method is demonstrated by analysis of the acid content of bornylacetate, bornylisovalerianate, α -acetyldigoxin, crystallized "aescin", valtrate and didrovaltrate, giving a recovery of 96.5–102%. Advantages and drawbacks —e.g. occurrence of "ghosting" after injection of impure samples— are discussed.

EINLEITUNG

Ester aliphatischer gesättigter und/oder ungesättigter C₂–C₆-Monocarbonsäuren kommen in einer grossen Anzahl von Pflanzen vor. So wurden z.B. verschiedene Komponenten in ätherischen Ölen¹, einzelne Asclepiadaceenbitterstoffe², Boragina-ceen- und Veratrum-Alkaloide², herzwirksame Glykoside³, Valepotriate⁴ und nicht zuletzt Saponine^{5–11} als Ester diverser kurzkettiger Monocarbonsäuren erkannt. Für die Strukturaufklärung solcher Verbindungen stellt die analytische Erfassung der qualitativen und quantitativen Säurezusammensetzung ein wichtiges Hilfsmittel dar.

Im Verlaufe eigener Untersuchungen an Estersaponinen¹² ergab sich die Notwendigkeit, ein zuverlässiges, einfaches und möglichst allgemein anwendbares gaschromatographisches Verfahren zu entwickeln, weil die bisher beschriebenen Methoden nur einen Teil der möglichen Carbonsäuren berücksichtigten^{8–11} oder auf

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Grund ungünstiger Trennleistungen quantitative Aussagen nur in begrenztem Masse zulassen¹³. Wegen der einfacheren Probenvorbereitung schien die direkte Analyse der freien Carbonsäuren aus wässriger Lösung am besten geeignet, obwohl bei dieser Vorgangsweise eine Reihe technischer Probleme —z.B. die Vermeidung von "Ghosting"— zu berücksichtigen war^{14,15}. Grosse Bedeutung kam der Auswahl der stationären Phase zu, da auch die ungesättigten Säuren (Dimethylacryl-, Angelika- und Tiglinsäure) bestimmt werden sollten, deren Retentionsverhalten im Rahmen früherer Arbeiten^{14–39} nicht untersucht wurde. Da einerseits Styrol-Divinylbenzol-Polymere, vor allem aber Chromosorb 101^{28,31} und andererseits flüssige Silikonphasen^{28,38} gute Trennergebnisse gezeigt hatten, ergab sich die Frage, ob ein durch Kombination beider resultierendes Gas-Liquid-Solid-Chromatographie-System auch die Analyse der ungesättigten Säuren erlaubt; dabei war zu hoffen, dass die zur Vermeidung von "Ghosting" empfohlene Sättigung des Trägergases mit Ameisensäure^{14,28} vermieden werden könnte. Eine weitere Steigerung der Trennleistung war durch Verwendung von mikrogepackten Säulen zu erwarten^{34,40,41}. Um auch Aussagen über die allgemeine Anwendbarkeit des neuen Verfahrens zu ermöglichen, wurden die Wiederfindungsrate bei der Analyse natürlicher Ester bekannten Säuregehaltes ermittelt, wobei die Verseifung unter möglichst schonenden Bedingungen erfolgte.

EXPERIMENTELLES

Gas-Chromatographie

Geräte. Gaschromatograph Perkin-Elmer F 33 mit Flammenionisationsdetektor; Integrator Perkin-Elmer SIP-1; Schreiber Perkin-Elmer 56 am logarithmischen Integratorausgang.

Packung. Unbelegtes Chromosorb 101 (100–120 mesh) (Johns-Manville, Denver, Colo., U.S.A.); Säule, Pyrex 6 ft. × 2 mm I.D.; Trägergas (N₂), 35 ml/min; Temperatur, Injektor-Detektor 175°, Ofen 160° (isotherm); Empfindlichkeit, 10 × 32.

Trennflüssigkeitsbelegungen auf Chromosorb 101. 6% DEGS, 6% EGA, 6% Carbowax 20M, 6% OV-210 und 6% OV-101; Säule, Pyrex 6 ft × 2 mm I.D.; Trägergasfluss und Temperatur wurden für jede Packung optimiert.

Bedingungen für quantitative Untersuchungen. Säule, Pyrex 2.4 m × 1 mm I.D.; Packung, 6% OV-210 auf Chromosorb 101 (100–120 mesh); Trägergas (N₂) 6 ml/min; Temperatur, Injektor/Detektor 225°, Ofen 170° (isotherm); Empfindlichkeit, 10 × 32.

Die Belegung von Chromosorb 101 erfolgte nach dem Verfahren von Williams⁴², die Säulenfüllung wurden unter vermindertem Druck und Anlegen von Ultraschall vorgenommen, das hintere Ende der Säule war mit einem kleinen Pfropfen silylierter Glaswolle verschlossen. Berechnung der Standardkorrekturfaktoren und Quantifizierung der Säuren in der üblichen Weise⁴³, die Reinheit der Testsubstanzen wurde bei der Angabe der Einwaagen berücksichtigt.

Substanzen

Vergleichssubstanzen. Essigsäure, Propionsäure, *n*-Propanol, Isobutanol, *n*-Butanol, Isoamylalkohol, *tert*.-Amylalkohol, Pentanol-2, Pentanol-3, Methyläthylketon und Isobutylmethylketon —jeweils höchster erhältlicher Reinheitsgrad— Merck (Darmstadt, G.F.R.); *n*-Buttersäure, Isobuttersäure, Isovaleriansäure, *n*-Valeriansäure, Isocaproensäure, *n*-Caproensäure, 3,3-Dimethylacrylsäure und Tiglin-

säure— jeweils höchster Reinheitsgrad— Fluka (Buchs, Schweiz). Angelikasäure wurde nach dem Verfahren von Buckles und Mock⁴⁴ aus Tiglinsäure hergestellt.

Alkalische Hydrolyse. Das Erwärmen der Reaktionsansätze erfolgte jeweils unter Rückflussschülung; Ansätze: 7 mg Bornylisovalerianat bzw. 15 mg Bornylacetat (Dragoco, Wien, Österreich) + 2 ml 10% methanolischer Kalilauge; 55 mg α -Acetyldigoxin + 7 ml 5% methanolischer Kalilauge; 190 mg "Aescin" (Fa. Dr. Madaus & Co.) + 6 ml Methanol + 2 ml 5% wässrige Kalilauge (1 h bei 95°); 9 mg Valtrat bzw. 8 mg Didrovaltrat + 2 ml 5% methanolischer Kalilauge; weitere Angaben siehe Anwendungsbeispiele.

ERGEBNISSE UND DISKUSSION

Trennung

Grundlage der vorliegenden Untersuchung war das Trennverhalten der gesättigten und ungesättigten C₂–C₆-Carbonsäuren auf unbelegtem Chromosorb 101. Bei optimalen Bedingungen konnten die Angaben von Ackman³¹ und Ottenstein und Bartley²⁸ bestätigt werden, allerdings wiesen die Paare Isovaleriansäure–2-Methylbuttersäure und Valeriansäure–Angelikasäure gleiche Retentionszeit auf und Capron-, Tiglin-, Isocapron- und 3,3-Dimethylacrylsäure waren unscharf getrennt (Tailing, breite Signale).

Die für die Trennung der kritischen Paare notwendige Selektivitätssteigerung der stationären Phase erschien am besten durch Belegung des Chromosorb 101 mit einer geeigneten Trennflüssigkeit erreichbar. Um einen möglichst grossen Polaritätsbereich zu erfassen, wählten wir DEGS, EGA, Carbowax 20M, OV-210 und OV-101; das bereits früher für die Analyse wässriger Carbonsäurelösungen empfohlene FFAP¹⁵ kam wegen der zu geringen Stabilität gegenüber Wasser³² nicht in Betracht. Zum Vergleich belegten wir Chromosorb 101 mit jeweils 6% der flüssigen Phasen—eine 6–8% Beladung hatte sich z.B. für Chromosorb 102 als optimal erwiesen⁴³—und stellten folgende Trenncharakteristika fest: OV-101 brachte die erwartete Verkürzung der Retentionszeiten⁴⁵, eine Veränderung der Elutionsfolge bzw. Verbesserung der Trennleistung im Vergleich zu unbelegtem Chromosorb 101 war nicht zu beobachten. DEGS und EGA ergaben eine ausgezeichnete Auftrennung der C₂–C₄-Säuren, Isocapron- und Tiglinsäure überlagerten jedoch; ausserdem zeigte sich starkes Tailing. Bessere Ergebnisse brachte der Einsatz von Carbowax 20M und OV-210; Valerian- und Angelikasäure wiesen nun eine Retentionszeitdifferenz von 0.9 min auf, für 3,3-Dimethylacryl-, Tiglin-, Isocapron- und Capronsäure ergab sich durch die deutliche Verminderung des Tailings eine verbesserte Auflösung; bei dem Paar 2-Methylbuttersäure–Isovaleriansäure deutete eine Schulter das Vorliegen zweier Verbindungen an. Nachdem die Silikonphase auf Grund ihrer Stabilität vorzuziehen war, wurde zunächst die beste Belegungskonzentration für OV-210 ermittelt. Das Optimum lag bei der bisher verwendeten 6% Beladung, womit eine Erhöhung der Trennkapazität nur durch Verlängerung der Säule und gleichzeitige Reduktion des I.D. zu erreichen war. In der Praxis erwiesen sich Säulen mit 1 mm I.D. am besten geeignet, weil sich die Füllung ohne grossen technischen Aufwand durchführen liess. Die entsprechenden Analysen zeigten eine deutliche Verbesserung der Trennleistung (Fig. 1 und 2): Valerian- und Angelikasäure waren nun für eine quantitative Bestimmung ausreichend getrennt, 2-Methylbutter- und Isovaleriansäure wiesen eine Retentionsdifferenz von

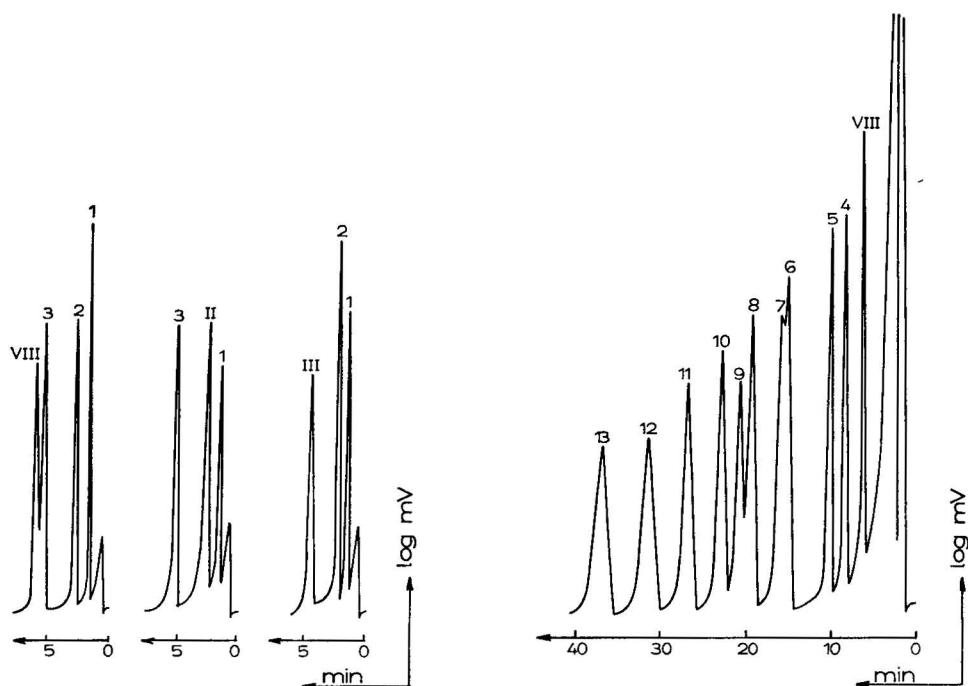


Fig. 1. Gaschromatographische Bestimmung von Essig- und Propionsäure aus wässriger Lösung unter Verwendung verschiedener innerer Standards; 1 = Ameisensäure, 2 = Essigsäure, 3 = Propionsäure; II = Isobutanol, III = *n*-Butanol, VIII = Pentanol-3.

Fig. 2. Gaschromatographische Bestimmung der C₄-C₆-Carbonsäuren aus wässrig-methanolischer Lösung; 4 = Isobuttersäure, 5 = *n*-Buttersäure, 6 = Isovaleriansäure, 7 = 2-Methylbuttersäure, 8 = *n*-Valeriansäure, 9 = Angelikasäure, 10 = 3,3-Dimethylacrylsäure, 11 = Tiglinsäure, 12 = Isocaproensäure, 13 = *n*-Capronsäure; VIII = Pentanol-3.

0.9 min auf und die Flächen wurden bei Vorliegen günstiger Mengenverhältnisse vom Integrator einzeln erfasst. Für den Nachweis von Essigsäure empfahl sich die Einspritzung wässriger Lösungen, da Methanol, aber auch Dichlormethan empfindlich störten. Unabhängig vom Lösungsmittel zeigten die Peaks der Säuren nur äusserst geringes Tailing.

Wie bereits einleitend erwähnt, war der Vermeidung von "Ghosting"-Effekten vor allem im Hinblick auf die Quantifizierung der Säuren besonderes Augenmerk zu schenken. Obwohl wir allen Probenlösungen einen Überschuss (mindestens 5%) Ameisensäure zusetzten und ausschliesslich Glassäulen mit "on column"-Einspritzung verwendeten, stellten wir zunächst bei nachfolgender Injektion von 1 μ l 5% wässriger Ameisensäure starkes "Ghosting" fest, wobei sich für die einzelnen Säuren exakt gleiche Retentionszeiten und Peakformen ergaben, wie bei normalen Analysen; die Adsorption musste also am Säulenkopf stattgefunden haben. Nach Entfernen der am Säulenanfang sitzenden Glaswolle (diese ist für den Verschluss von Säulen mit 1 mm I.D. nicht notwendig) fanden wir nach sechsmaliger Injektion eines Testgemisches —es wurden von jeder Säure jeweils 3 μ g dosiert— keinerlei Anzeichen von "Ghosting". Dies bestätigte die Ergebnisse von Geddes und Gilmour⁴⁶ und zeigte die Eignung der Säulenpackung (6% OV-210 auf Chromosorb 101) für die quantitative Analyse

kurzkettiger Carbonsäuren; dadurch erübrigte sich auch die Zumischung von Ameisensäure zum Trägergas.

Quantifizierung

Bei der Untersuchung biologischen Probenmaterials war mit der Ablagerung bzw. Zersetzung verschiedener Begleitstoffe im Injektionsbereich der Säulen zu rechnen, wodurch unerwünschte Adsorption der freien Carbonsäuren eintreten kann⁴⁶. Um daraus resultierende systematische Fehler auszuschliessen, empfahl sich die Verwendung eines inneren Standards mit geringer Adsorptionstendenz. Am besten geeignet erschienen im Handel erhältliche, niedermolekulare Ketone, aber auch Alkohole, da das früher vorgeschlagene Cyclohexanon³¹ vor allem für die Analyse der Essig- und Propionsäure aus wässriger Lösung zu lange Retentionszeiten aufwies. Folgende Verbindungen wurden auf ihre Eignung geprüft: *n*-Propanol (I), Isobutanol (II), *n*-Butanol (III), Isoamylalkohol (IV), *n*-Amylalkohol (V), *tert*.-Amylalkohol (VI), Pentanol-2 (VII), Pentanol-3 (VIII), Methyläthylketon (IX) und Isobutylmethylketon (X). Während IX im Lösungsmittelpack lag, überlappten die Peaks von I und II mit Essigsäure, III, VI, VII und VIII mit Propionsäure und IV, V und X mit Isobuttersäure. Es waren daher für die quantitative Bestimmung je nach Analysenproblem verschiedene Standards vorzusehen: Proben, die keine oder nur relative geringe Mengen Propionsäure enthielten, liessen sich am besten unter Verwendung von VIII bestimmen. Bei Vorliegen grosser Mengen Propionsäure bot sich als Standard V an, wobei allerdings Isobuttersäure störte. Selbstverständlich konnte die rasche Analyse der Proben, in denen nur Essigsäure zu erwarten war, mittels III und solcher mit Propionsäure unter Anwendung von II als Standard vorgenommen werden. Um die allgemeine Anwendbarkeit des Verfahrens zu gewährleisten, wurden für alle diese Möglichkeiten Eichungen durchgeführt (Fig. 1 und 2; Tabelle I).

Die in Tabelle I angegebenen relativen Standardabweichungen brachten im geprüften Konzentrationsbereich für alle Carbonsäuren eine wesentlich bessere Analysengenauigkeit als mit unbelegtem Chromosorb 101¹⁴. Da für alle Säuren Regressionsgerade resultierten, die durch den Nullpunkt verliefen und die Ansprechempfindlichkeiten für äquivalente Gewichte der gesättigten Säuren im Vergleich zu früheren Angaben⁴⁴ im Mittel nur um 5.7% differierten, war die Eignung des vorgeschlagenen GLSC-Systems für quantitative Analysen bewiesen.

Anwendungsbeispiele

Die Möglichkeit der direkten Analyse wässriger bzw. wässrig-methanolischer Lösungen der freien Carbonsäuren hatte gleichzeitig auch eine Vereinfachung der Probenvorbereitung zur Folge, da sich zeitaufwendige Extraktionsprozesse mit organischen Lösungsmitteln erübrigten. Der Säuregehalt aller natürlichen Ester, welche wir als Testsubstanzen heranzogen, konnte nach folgendem, einfachem Verfahren ermittelt werden: Man löst die Probe in Wasser, Wasser-Methanol oder Methanol und versetzt mit dem entsprechenden Hydrolysereagens. Nach der Verseifung wird das Methanol möglichst vollständig entfernt, aus einer Stammlösung der innere Standard zugesetzt und in genügendem Überschuss mit Ameisensäure angesäuert. Diese Lösung dient zur gaschromatographischen Bestimmung von Essigsäure und/oder Propionsäure. Nach Verdünnen mit Methanol auf das doppelte Volumen (Löslichkeit der C₆-Säuren) können die C₄-C₆-Carbonsäuren analysiert werden.

Die Bestimmung des Isovaleriansäuregehaltes von Bornylisovalerianat ergab

TABELLE I

STANDARDKORREKTURFAKTOREN (F) UND RELATIVE STANDARDABWEICHUNGEN (s_r , rel.) BEI DER ANALYSE VON TESTGEMISCHEN MIT VERSCHIEDENEN INNEREN STANDARDS

Es wurden jeweils 0,2–0,8 μl dosiert; Essigsäure und Propionsäure in wässriger Lösung, alle anderen Säuren in Methanol–Wasser (1:1), wobei sich gleiche Korrekturfaktoren wie in Dichlormethanolösung ergaben.

Säure	Konzentrationsbereich ($\mu\text{g}/\mu\text{l}$)	Standard	Zuwaage Standard ($\mu\text{g}/\mu\text{l}$)	F	s_r , rel. (%)
Essigsäure	0.34–3.4	<i>n</i> -Butanol	0.32	3.42	± 2.22
Essigsäure	0.66–3.3	<i>n</i> -Amylalkohol*	1.95	3.66	± 4.24
Propionsäure	0.65–3.3	<i>n</i> -Amylalkohol	1.95	1.98	± 2.67
Propionsäure	0.64–3.6	Isobutanol	0.32	1.83	± 3.89
Isobuttersäure	0.64–3.2	Pentanol-3	2.03	1.54	± 2.22
<i>n</i> -Buttersäure	0.66–3.3	Pentanol-3	2.03	1.58	± 1.62
2-Methylbuttersäure**	0.80–4.0	Pentanol-3	2.03	1.36	± 2.94
Isovaleriansäure	0.62–3.1	Pentanol-3	2.03	1.36	± 2.80
<i>n</i> -Valeriansäure	0.64–3.2	Pentanol-3	2.03	1.40	± 2.14
Angelikasäure	0.52–1.6	Pentanol-3	2.03	1.45	± 1.61
3,3-Dimethylacrylsäure	0.65–3.2	Pentanol-3	2.03	1.47	± 2.45
Tiglinsäure	0.66–3.3	Pentanol-3	2.03	1.57	± 2.80
Isocaproensäure	0.64–3.2	Pentanol-3	2.03	1.44	± 2.36
<i>n</i> -Capronsäure	0.67–3.3	Pentanol-3	2.03	1.42	± 1.97

* *n*-Amylalkohol und Pentanol-3 wiesen die gleiche molare Ansprechempfindlichkeit auf.

** 2-Methylbuttersäure und Isovaleriansäure wurden aus unterschiedlichen Testgemischen bestimmt.

nach zweistündiger Verseifung mit 10% methanolischer Kalilauge bei 90° eine Wiederfindungsrate von 99%. Bei der Analyse des Essigsäureanteils in Bornylacetat fanden wir bei einer Reaktionstemperatur von 50° 96.5% und bei 90° 98% der vorausberechneten Menge.

Zur Ermittlung des Essigsäuregehaltes von α -Acetyldigoxin waren wesentlich mildere Verseifungsbedingungen möglich. Bereits nach einstündigem Erwärmen mit 5% methanolischer Kalilauge auf 45° wiesen wir 98% des berechneten Säuregehaltes nach.

Estersaponine behalten meist auch nach Verseifung ihre oberflächenaktiven Eigenschaften, wodurch bei Ausschüttelung mit organischen Lösungsmitteln stabile Emulsionen entstehen können. Durch die Möglichkeit der direkten Einspritzung wässriger Lösungen war es mit dem neuen Verfahren möglich, dieses Problem zu umgehen. Bei der Untersuchung von kristallisiertem "Aescin" wiesen wir Essig-, Isobutter-, 2-Methylbutter-, Angelika- und Tiglinsäure nach; die quantitative Auswertung ergab ein molares Verhältnis von 10:1:1:4:5.5, welches die C₄–C₅-Säuren betreffend ziemlich genau mit früheren Angaben¹¹ übereinstimmte, der um 10% höhere Essigsäureanteil lässt sich auf die verbesserte Probenvorbereitung zurückführen. Aus den entsprechenden Chromatogrammen (Fig. 3) ist ersichtlich, dass die weitgehende Entfernung des bei der Hydrolyse verwendeten Methanols eine verbesserte Auflösung von Essigsäure- und Lösungsmittelpeak zur Folge hat.

Die Grenzen des vorgeschlagenen Verfahrens wurden bei der Bestimmung des

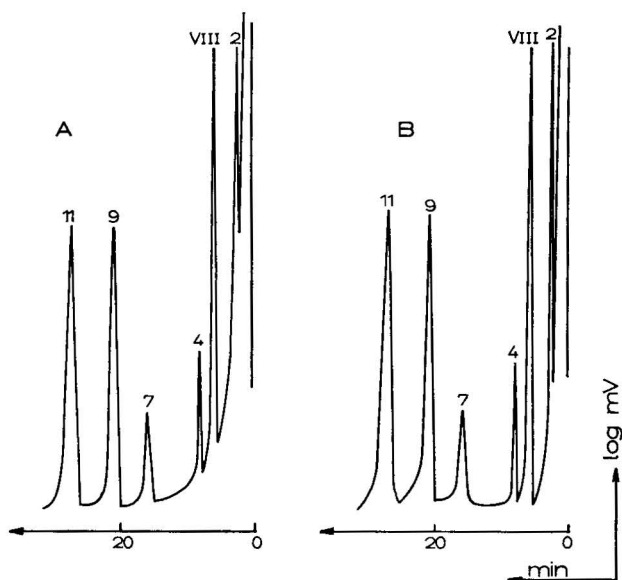


Fig. 3. Gaschromatographische Bestimmung der in Aescin enthaltenen Carbonsäuren; A = wässrigmethanolische Lösung, B = wässrige Lösung (Methanol weitgehend entfernt); 2 = Essigsäure, 4 = Isobuttersäure, 7 = 2-Methylbuttersäure, 9 = Angelikasäure, 11 = Tiglinsäure; VIII = Pentanol-3.

Säuregehaltes von Valepotriaten deutlich. Zunächst erhielten wir bei Valtrat sowie auch bei Didrovaltrat eine Wiederfindungsrate von 99% Essigsäure und 102% Isovaleriansäure. Nach mehreren Analysen trat jedoch bei Nachspritzen von Ameisensäure deutliches "Ghosting" auf, welches durch Kondensate in der Einspritzzone der Säule verursacht wurde. Die Einspritzlösungen waren auch nach schonendster Verseifung durch Reaktionsnebenprodukte, die sich mit organischen Lösungsmitteln nicht extrahieren liessen, tiefbraun gefärbt. Obzwar sich nach Reinigung und Neu-füllen des Säulenansatzes immer zufriedenstellende Wiederfindungsraten erzielen liessen, machte dieses Beispiel deutlich, dass bei der direkten analytischen Erfassung der freien C_2 - C_6 -Carbonsäuren aus biologischen Material eine ständige Prüfung des gaschromatographischen Systems durch Nachspritzen von wässriger Ameisensäure und laufende Kontrolle der Standardkorrekturfaktoren zu erfolgen hat. Unter dieser Voraussetzung erfüllt das neue Verfahren alle Anforderungen bezüglich Genauigkeit und Einfachheit. Der technische Aufwand ist relativ gering, da die erforderlichen Glassäulen an jeden Gaschromatographen, der "on column"-Injektion erlaubt, angeschlossen werden können.

ZUSAMMENFASSUNG

Eine neues Gas-Liquid-Solid-Chromatographie-Verfahren (Stationäre Phase, 6% OV-210 auf Chromosorb 101) erlaubt unter Verwendung von mikropackten Glassäulen ohne Sättigung des Trägergases mit Ameisensäuredämpfen die Trennung und Bestimmung von Essig-, Propion-, Isobutter-, *n*-Butter-, 2-Methylbutter-, Isovalerian-, *n*-Valerian-, Isocapron-, *n*-Capron-, 3,3-Dimethylacryl-, Angelika- und

Tiglinsäure aus natürlich vorkommenden Estern. Die Quantifizierung erfolgt unter Verwendung von innerem Standard, die untere Grenze des linearen Bereichs liegt bei 0.1 µg/Injektion. Die allgemeine Anwendbarkeit der neuen Methode wird an Hand der Analyse des Säuregehaltes von Bornylacetat, Bornylisovalerianat, α -Acetyldigoxin, kristallisiertem "Aescin", Valtrat und Didrovaltrat demonstriert; die Wiederfindungsraten liegen zwischen 96.5 und 102%. Vor- und Nachteile —z.B. das Auftreten von "Ghosting" nach Injektion stark verunreinigter Proben— werden diskutiert.

DANK

Für die Überlassung von Vergleichssubstanzen danken wir der Fa. Dragoco, Wien, Österreich, und Herrn Dr. P. W. Thies, Kali-Chemie, Pharma, Hannover, B.R.D.

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CHROM. 12,445

RAPID ANALYSIS OF CHLORINATED DIOXINS IN COMPLEX ORGANIC MIXTURES BY APPLICATION OF A GAS CHROMATOGRAPHY–MASS SPECTROMETRY–CALCULATOR SYSTEM WITH USER-DEVELOPED SOFTWARE

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SUMMARY

A procedure for the rapid analysis of chlorinated dioxins in incinerator fly ash samples by gas chromatography–mass spectrometry (GC–MS) is presented. After Soxhlet extraction of the fly ash with benzene and a 2000-fold sample concentration, the extracted sample is analyzed by GC–MS without further sample treatment. Development of software has permitted ion abundance data to be stored on a single floppy disk for later generation of the total ion abundance plot and as many as six mass chromatograms, in addition to the mass spectra of eluting components. Qualitative and quantitative data for various polychlorinated dibenzo-*p*-dioxins (PCDD) can be obtained from a single GC–MS run. By comparison with the peak height of a 1,2,3,4-tetrachlorodibenzo-*p*-dioxin standard, concentrations of various PCDD isomers were obtained for two different fly ash samples. Estimated sensitivity is 230 pg for the tetrachlorodibenzo-*p*-dioxins.

INTRODUCTION

Many analytical methods concerned with the analysis of toxic organic compounds found in the environment have been reported^{1–4}. For such applications, the primary analytical tools employed are gas chromatography (GC) and GC–mass spectrometry (MS). Unfortunately, many of the methods reported involve lengthy extraction procedures followed by complicated sample clean-up steps before GC and GC–MS analyses are carried out. The GC–MS instrumentation is usually a large and expensive computerized system.

Recently, a rapid analytical procedure was reported for the analysis of complex

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organic mixtures associated with atmospheric aerosols which eliminated the sample clean-up steps before analysis and used a relatively simple GC-MS-calculator system⁵. This technique has been applied to the analysis of the organic material extracted from fly ash samples collected from municipal incinerators⁶. Among the many compounds detected were various polychlorinated dibenzo-*p*-dioxin (PCDD) isomers. A modification of this procedure combined with special computer programs specifically for the rapid quantitative analysis of separated PCDD isomers extracted from fly ash samples is presented here.

The procedure consists of Soxhlet extraction of the fly ash with benzene followed by a 2000-fold sample concentration. The condensate is analyzed by GC-MS employing a highly efficient chromatographic packing without further sample treatment. An important aspect of this method is the development of software to control the operation of a GC-MS-calculator system which enabled both qualitative and quantitative analysis of the tetra- (TCDD), penta-, hexa-, hepta- and octachloro-dibenzo-*p*-dioxin (OCDD) isomers to be obtained from a single GC-MS run⁷. Analysis can be accomplished even for samples in which dioxin concentrations are very low compared to concentrations of other compounds which co-elute with the various dioxin isomers.

EXPERIMENTAL

Sample collection and storage

Fly ash samples were obtained from a municipal incinerator located in Southern Ontario. Kilogram quantities of grab samples from an electrostatic precipitator were supplied by Dr. A. Foldes of the Ontario Ministry of the Environment. Replicate samples were shipped and stored at ambient temperature in glass containers with metal screw-caps. Drying of the samples before extraction was not necessary.

Sample preparation

Approximately 20-g samples were weighed and placed into medium porosity glass fritted extraction thimbles. Overnight extractions (*ca.* 16 h) were performed in a Soxhlet extraction apparatus using 200 ml of benzene ("distilled-in-glass" grade, Caledon Labs., Georgetown, Canada). Each sample was extracted in replicate, as well as a procedure blank conducted using the assembled Soxhlet apparatus with 200 ml of benzene solvent and without a sample. The sample extract was condensed to about 0.3 ml by rotary evaporation under aspirator vacuum. The condensate was transferred with rinsing to a 1.0-ml graduated Reacti-vial (Pierce, Rockford, Ill., U.S.A.) equipped with screw-cap and PTFE liner. A final volume of 100 μ l was achieved by passing a gentle stream of high purity nitrogen (Linde, 99.995%) over the top of the Reacti-vial. Vials were stored in a freezer at -10 to -15° . All glassware was cleaned in an ultrasonic bath containing a 2% aqueous solution of Alconox detergent for 30 min, rinsing with deionized water, then heating in a laboratory oven for 1 h at 300° .

Analysis by GC-MS

A Hewlett-Packard 5992A GC-MS-calculator system equipped with single floppy disk, *x-y* plotter and 2 m $\times$ 2 mm I.D. glass column was used for GC-MS analysis. The chromatographic column was packed with a high-performance material

called Aue packing⁸. Chromatographic conditions were: initial temperature, 90°; program rate, 4°/min; final temperature, 250° held for 15 min or less; injection port temperature, 250°; helium carrier flow, 40 ml/min. The quadrupole mass spectrometer was repetitively scanned from 500 a.m.u. to 40 a.m.u. at 330 a.m.u./sec. Mass spectra taken at the top of eluting GC peaks were saved on floppy disk, as were mass spectra taken at the lowest valley between two peaks for later background subtraction. A 1,2,3,4-TCDD standard was run to provide quantitation. The mass spectrometer operating conditions were optimized each day using the manufacturer-supplied program AUTOTUNE. A silicone rubber membrane separator was employed as the GC-MS interface.

Generation of mass chromatograms

The manufacturer supplied GC-MS software did not permit storage of mass chromatogram and total ion current (TIC) data as well as the mass spectra. To conduct these analyses it was necessary to create a program, called Dual-Mode, capable of storing mass spectra, TIC ion data and mass chromatogram ion data in real-time on a single floppy disk⁷. Ion abundances for as many as six ions are extracted from each mass scan, stored on floppy disk and plotted later as mass chromatograms. This differs from the selected ion monitoring (SIM) technique, in which the mass spectrometer is consecutively tuned only to each of the ions selected by the user. Although the SIM technique offers greater sensitivity, no mass spectra can be generated. A separate program was written to plot results on the *x-y* plotter after completion of the run. Details of this software are available from the authors.

RESULTS AND DISCUSSION

By proper choice of the ions to be monitored, the technique of mass chromatography can be used to selectively detect specific compounds or compound classes. For this study, mass chromatograms were generated by Dual-Mode for the characteristic ions at *m/e* 321.9, 355.9, 389.8, 425.8 and 459.7; which permit detection of groups of isomers of the tetra-, penta-, hexa-, hepta- and octachlorodibenzo-*p*-dioxins, respectively.

Fig. 1 shows the TIC for the analysis of a fly ash sample along with the associated mass chromatograms for various chlorinated dioxin isomers. A procedure blank which was run immediately prior to this sample gave straight line plots for the mass chromatograms of the dioxin isomers, indicating that no detectable background compounds were present which possessed interfering ions for this analysis.

The five groups of peaks seen in the mass chromatogram plots of Fig. 1 increase in retention time by fairly constant increments from the *m/e* 321.9 ion, which is characteristic of TCDD, to the *m/e* 459.7 ion, which is characteristic of OCDD. This retention behavior is consistent with a series of compounds having similar structures but differing in one chlorine atom from one group of isomers to the next. Also it is important to note that only two distinct peaks are observed for *m/e* 425.8 ion, characteristic of heptachlorodibenzo-*p*-dioxins, which have only two isomers; and one peak is observed for *m/e* 459.7 ion, characteristic of OCDD which has no isomers. The retention time of the 1,2,3,4-TCDD standard coincided with the retention time observed for a sample peak of 22.5 min detected in the mass chromatogram of the *m/e*

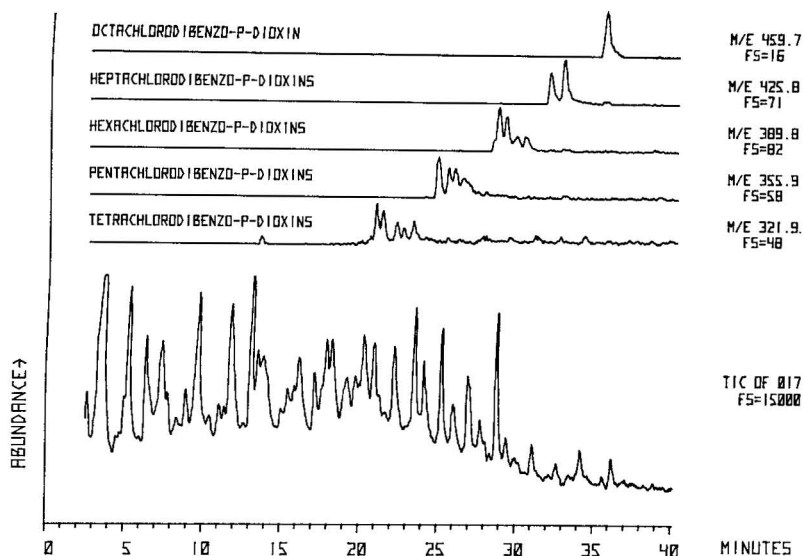


Fig. 1. Plot of TIC and five mass chromatograms from a Dual-Mode analysis of a fly ash sample. Injection volume was $3.1 \mu\text{l}$. The mass chromatograms are identified by a particular m/e value which corresponds to one set of PCDD isomers.

321.9 ion, characteristic of TCDD isomers. This is shown in Fig. 2, which is a comparison plot of the 1,2,3,4-TCDD standard and the mass chromatogram from Fig. 1 of the m/e 321.9 ion.

Fig. 1 shows that the various chlorinated dioxin isomers can be detected even in the presence of large amounts of other compounds which elute at the same or similar retention times. PCDD concentrations are low compared to many of the other

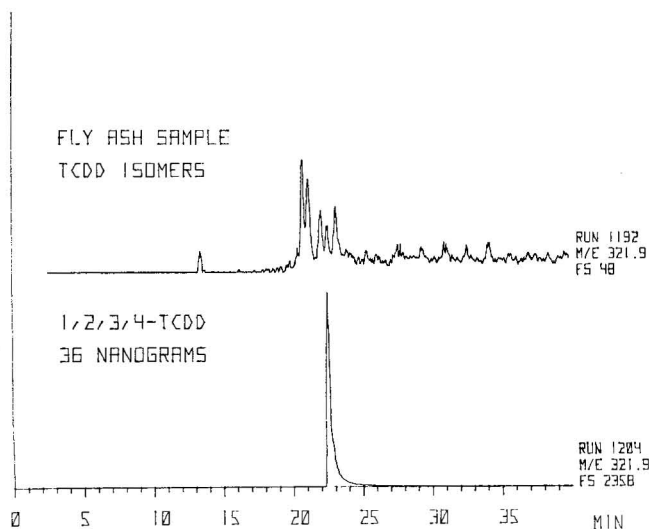


Fig. 2. Comparison between the mass chromatograms for m/e 321.9 from Fig. 1 and from a 1,2,3,4-TCDD standard run under identical conditions.

components shown in the TIC, however they are easily detected, since the ions which were chosen for the generation of mass chromatograms are free of interferences for this sample. The resolution observed for the isomers, in particular for isomers of TCDD, demonstrates the separation performance of the chromatographic column employed.

Fig. 3 presents the TIC and mass chromatograms of the dioxins for a second fly ash sample. Even though the total organic compounds detected are at very low levels compared to those in Fig. 1, it is still possible to detect selectively the various PCDD isomers. The relative patterns of the various isomers are almost identical to the patterns seen in Fig. 1.

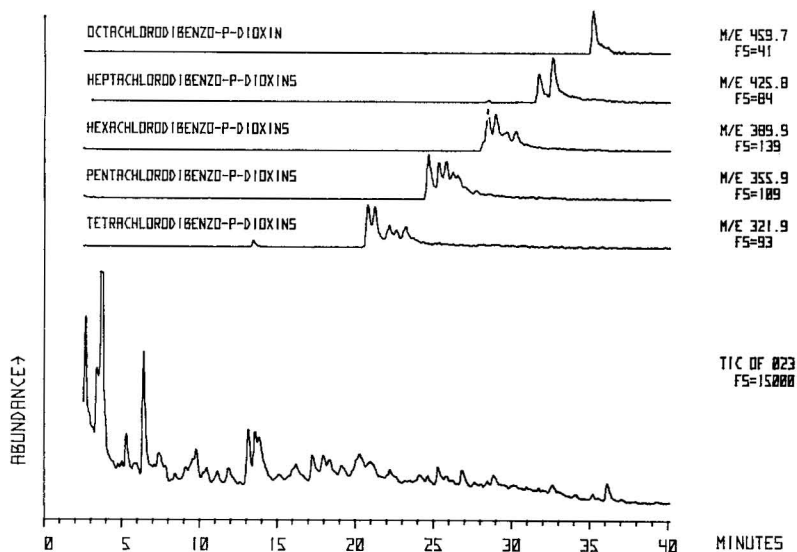


Fig. 3. Plot of TIC and five mass chromatograms for a second fly ash sample. Injection volume was 1.2 μ l. Various PCDD isomers are detected even though the total organic loading is much less than for the sample shown in Fig. 1.

Relative concentrations of PCDD may be determined by comparing the full-scale abundance values produced during the Dual-Mode run. By comparing these values given in Fig. 1, it is observed that the hexachlorinated isomers are of greater abundance than the other PCDD isomers for this sample, followed by the hepta-, penta-, tetra- and octachlorinated species. In Fig. 3 a different pattern of concentrations is observed as the hexachlorinated species are the most abundant, followed by the penta-, tetra-, hepta- and octachlorinated dioxins. Each set of PCDD isomers is of greater abundance for the sample shown in Fig. 3 than for the sample in Fig. 1.

By comparing peak heights from Figs. 1 and 3 to that of the 1,2,3,4-TCDD standard (Fig. 2), quantitation of the various PCDD isomers was calculated assuming the same response factor for all isomers. These estimates are presented in Table I.

Since there may exist other compounds which possess ions in their mass spectra having m/e values that correspond to the m/e values chosen as indicating the presence of the various PCDD compounds, it is necessary to have the complete mass spectrum

TABLE I

ESTIMATED CONCENTRATIONS OF PCDD ISOMERIC GROUPS IN FLY ASH SAMPLES

Based on peak height comparison of individual PCDD peaks from the mass chromatograms of Figs. 1 and 3 with the 1,2,3,4-TCDD standard in Fig. 2 run under the same conditions. A relative response factor of 1 is assumed for all isomers.

Isomeric group	Total concentration of isomer groups (ng/g)	
	Sample 1 (Fig. 1)	Sample 2 (Fig. 3)
Tetrachlorinated dioxins	4.2	19
Pentachlorinated dioxins	4.9	27
Hexachlorinated dioxins	5.1	24
Heptachlorinated dioxins	3.2	8.8
Octachlorodibenzo- <i>p</i> -dioxin	0.42	2.7

of each compound for positive identification. The advantage of the Dual-Mode run over the more sensitive SIM technique is that mass spectra of the detected components can be retrieved from disk and compared to mass spectra of standard compounds. Specific identification of individual isomers can then be made by matching retention times as in Fig. 2.

Fig. 4 shows the mass spectra of five PCDD compounds ranging from the tetrachlorinated to the octachlorinated species which were all obtained during a single

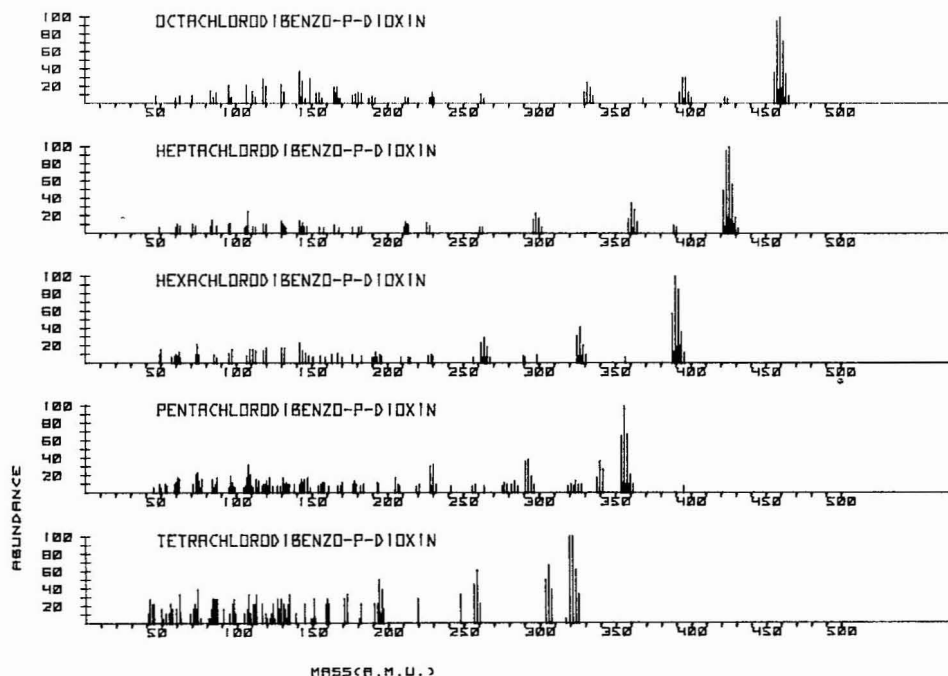


Fig. 4. Mass spectra of tetra-, penta-, hexa- and octachlorinated dibenzo-*p*-dioxins obtained from a single Dual-Mode run of a fly ash sample.

Dual-Mode run of a fly ash sample. The TCDD spectrum was obtained from a very low abundance peak, however the characteristic isotope pattern is still observable. Isotope ratios are not ideal due to the low abundance of the TCDD peak.

Limits of detection can be obtained from data given in Fig. 2. By choosing a noise threshold of 15 counts (full scale), the standard run corresponds to a detection limit of 230 picograms for 1,2,3,4-TCDD. The choice of 15 counts full scale as a noise limit is based on the background response seen in Fig. 2 beginning at a retention time of about 25 min. Lower detection limits can be obtained for some of the other PCDD isomers which have lower levels of background contamination.

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CHROM. 12,440

DETERMINATION OF ORGANOPHOSPHORIC AND ORGANOPHOSPHOROTHIOIC ACIDS IN AQUEOUS SOLUTIONS BY ION CHROMATOGRAPHY

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SUMMARY

An ion chromatographic procedure is described for the separation and determination of methyl and ethyl phosphates, phosphorothioates and phosphoric acid in aqueous solutions at the part per million level. The separations are achieved on columns of low capacity anion-exchange resin with sodium carbonate-bicarbonate eluent. Detection is accomplished by a combination of conductivity and UV absorbance at 210 nm. The UV detector gives more specificity and sensitivity for organophosphorothioates, while discriminating against a strong chloride interference with the conductimetric detection of the dialkylated organophosphorothioic acids.

INTRODUCTION

In the past gas chromatographic (GC) methods for measurement of organophosphoric and organophosphorothioic acids in waste brines and foodstuffs¹ have been employed. The acids must be isolated from the water and derivatized with diazomethane before analysis, however. Thin-layer chromatography (TLC) has also been a popular technique for determining the acids²⁻⁴ but it suffers from many disadvantages, non-quantitative results, extraction is required and difficulties with visualization of the developed TLC plate.

Recently an ion chromatographic determination of dibutylphosphate in water was reported⁵. We also were inspired by the development of ion chromatography and achieved an ion chromatographic procedure for determining the methyl and ethyl phosphates and phosphorothioates in water. However, the major components of interest, diethylphosphorothioic acid (DEPT) and dimethylphosphorothioic acid (DMPT) were obscured by chloride in the samples of interest.

Since organophosphates are not detectable by UV absorption it was decided to retain the conductivity detector for this determination and to add a UV detector to the conductivity cell outlet. This would take advantage of the strong UV absorption properties of the phosphorothioic acids and discriminate against the chloride interference.

EXPERIMENTAL

Apparatus

A schematic of the analytical system used is shown in Fig. 1. Three 300×2.8 mm glass columns packed with a low-capacity anion-exchange resin agglomerated on 180–230 mesh surface-sulfonated styrene divinyl benzene, 0.011 mequiv./g capacity were used. One 300×2.8 mm glass column, packed with Dowex AG 50W-X16 high-capacity cation-exchange resin, 200–400 mesh in the hydrogen form was employed. Sodium carbonate–sodium bicarbonate, 0.0012 *M* each in distilled deionized water at 1.0 ml/min flow-rate was used as eluent. A Modern Metalcraft BM-2 conductivity cell and a Perkin-Elmer LC-55 UV detector at 210 nm were used.

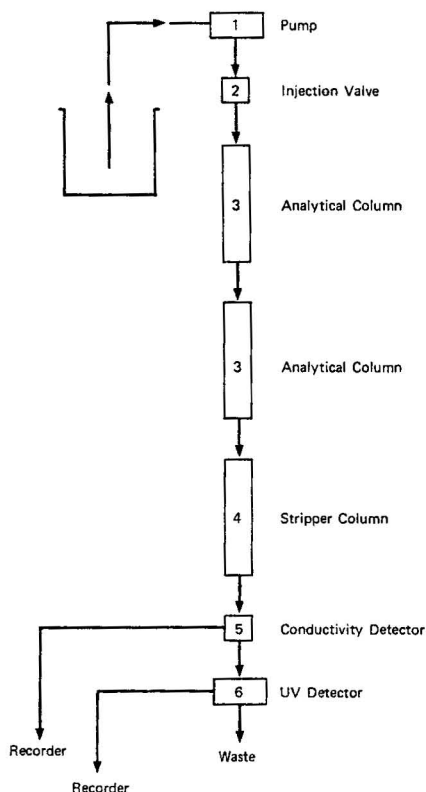


Fig. 1. Schematic diagram of apparatus.

The sample size was 170 μ l. A small 100×2.8 mm glass column packed with cation-exchange resin was connected to the outlet of the analytical system to eliminate bubble formation from the carbonic acid in the eluent.

Reagents

Analytical-grade sodium carbonate and sodium bicarbonate were used to prepare the mobile phase. For regeneration of the stripping column, 1.0 *N* reagent-

grade nitric acid was used. The stripper was regenerated for about 20 min and then washed with deionized water until the column effluent was neutral (≈ 15 min).

Standards

The standards and other reagents used, when commercially available, were technical grade or better. Three, however, were prepared in the Agricultural Synthesis Group at Dow. They were potassium O,O-diethylphosphate (KDEP), potassium O,O-diethylphosphorothioate (KDEPT) and potassium O,O-dimethylphosphorothioate (KDMPT). They were analysed by phosphorus ^{31}P nuclear magnetic resonance (NMR) and by infrared (IR) spectrometry for impurities and verification of structure. All three standards showed traces of water by IR, but only in the case of KDMPT was any other impurity detected. From the NMR spectrum the KDMPT standard appears to contain about 4% of what is suspected to be O-methyl-O-ethylphosphorothioate. The monoethylphosphoric (MEP) and monomethylphosphoric (MMP) acids were obtained commercially and found by titration with standard base and by NMR to be mixtures of roughly equal concentrations of the corresponding mono- and disubstituted organophosphoric acids. The structures of the compounds of interest are shown in Fig. 2.

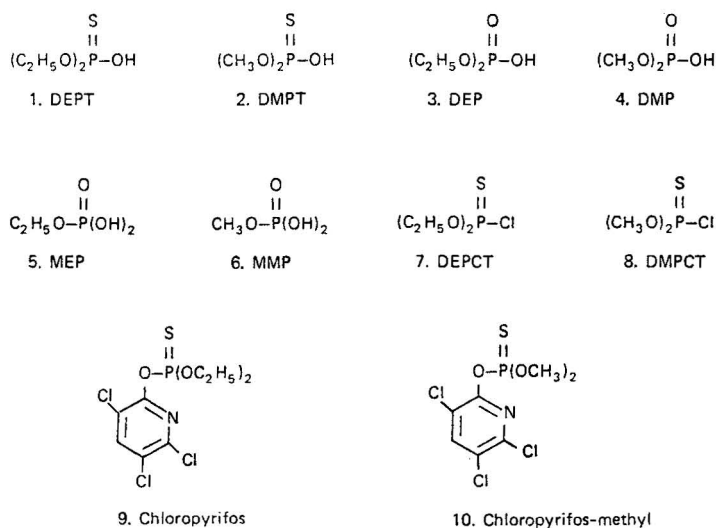


Fig. 2. Structures of components.

RESULTS AND DISCUSSION

In the original work three separating columns of an anion-exchange resin prepared in our laboratories were used. In a later work, analysis of chloropyrifos-methyl, a Dow product, was performed using only one separating column obtained from Dionex (Sunnyvale, Calif., U.S.A.). The obtained separations were comparable to those obtained in the original work. However retention times with the Dionex column were much shorter. Chloride still proved to be a major interference for the detection of DEPT and DMPT by conductivity and the UV detector was required.

Fig. 3 is a UV spectrum of KDEPT in water, obtained with a Cary 17 spectrophotometer. From the spectrum the 210 nm operating wavelength for the UV detector was chosen.

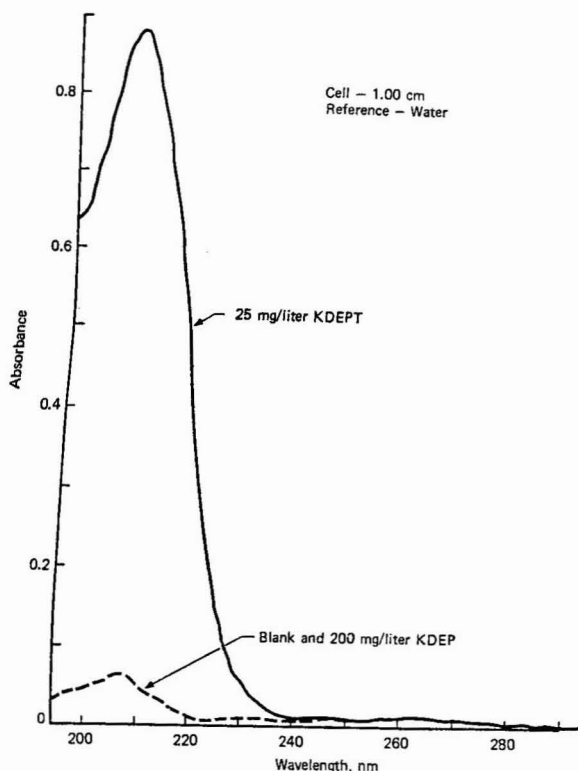


Fig. 3. UV absorption spectrum of potassium O,O-diethylphosphorothioate.

In Figs. 4 and 5 the dual chromatograms for the ethyl and methyl substituted phosphorus species are shown. Chloride at 2.5 ppm has been added in one case to demonstrate the interference observed for the phosphorothioic acids using the conductivity cell response. The eluent strength was chosen as a compromise between short retention times for disubstituted phosphates and long retention times for mono-substituted phosphates.

When water is injected into the system a negative response results near the void volume of the column, and hence near the elution time of the disubstituted phosphates. This makes quantitation for them difficult. The negative peak is the result of a momentary decrease in the carbonic acid concentration in the eluent, and can be eliminated by preparing the standards in the eluent or by adding to carbonate free water samples 1.0% of 100-fold concentrated eluent. Table I lists the estimated detection limits that would have given signal-to-noise ratio of 2.5 on the day the particular standards were run. By optimizing the elution times for anyone particular species, better detection limits could be achieved.

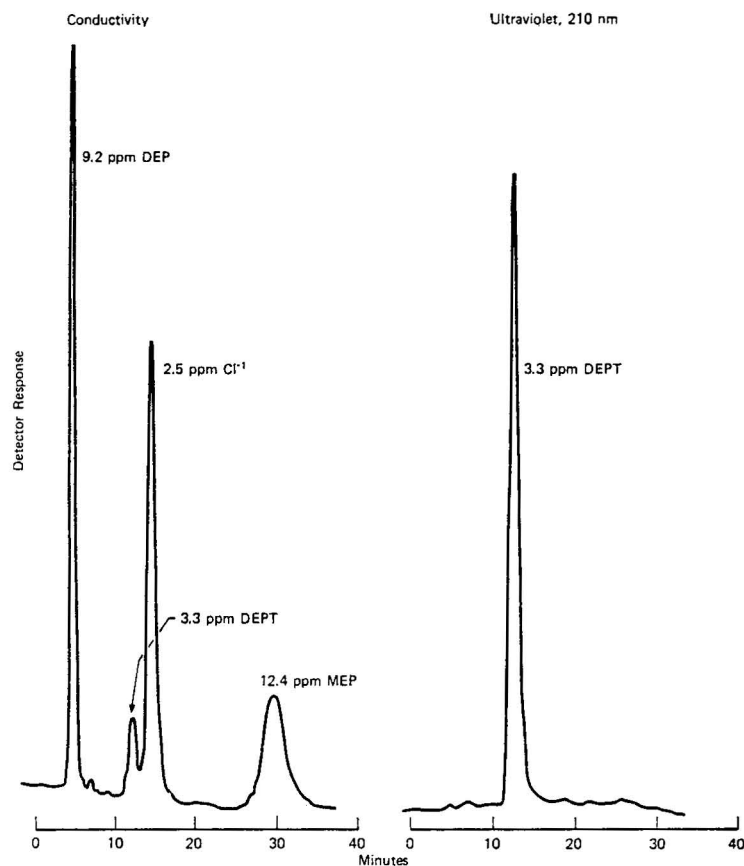


Fig. 4. Dual detector chromatograms of ethyl substituted phosphorus species.

TABLE I

ESTIMATED DETECTION LIMITS

Signal-to-noise ratio, 2.5.

Compound	Detector	
	Conductivity (mg/l)	UV 210 nm (mg/l)
Diethylphosphate	0.6	—
Dimethylphosphate	0.4	—
Diethylphosphorothioate	0.9	0.2
Dimethylphosphorothioate	0.9	0.2
Diethylphosphate	2.2	—
Monoethylphosphate	1.6	—

Table II lists the retention times and capacity factors for the organophosphorus species tested and for some typical compounds that could be expected in water samples. It is not possible to separate adequately the mono- and dimethylphosphates

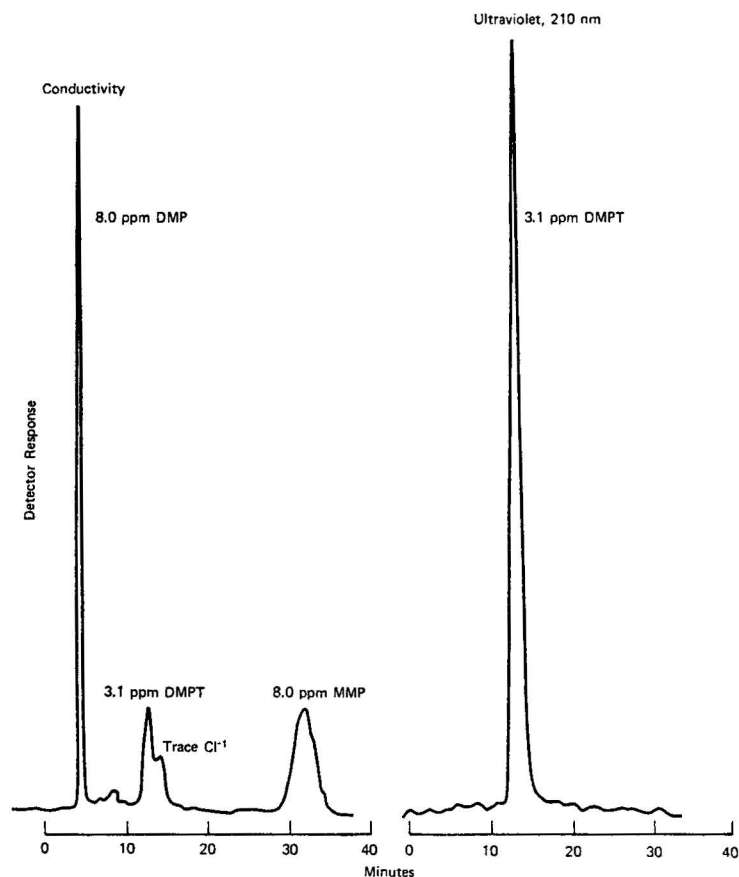


Fig. 5. Dual detector chromatograms of methyl substituted phosphorus species.

TABLE II

ELUTION TIMES FOR ORGANOPHOSPHATES, PHOSPHOROTHIOATES AND SOME POTENTIAL INTERFERENTS

Compound	Elution time (min)	Capacity factor	UV response
Diethylphosphate	4.4	0.1	No
Dimethylphosphate	4.4	0.1	No
Acetate	5.4	0.4	Yes
Glycolate	5.4	0.4	Yes
Formate	6.3	0.7	Yes
Monochloroacetate	8.8	1.3	Yes
Diethylphosphorothioate	11.8	2.1	Yes
Dimethylphosphorothioate	12.5	2.3	Yes
Chloride	14.0	2.7	No
Dichloroacetate	23	5.0	Yes
Monoethylphosphate	28	6.3	No
Monomethylphosphate	31	7.1	No
Nitrate	≈ 46	11	—
Orthophosphate	> 60	> 15	No
Sulfate	> 60	> 15	No

from their corresponding ethyl phosphates, but when all are present at similar levels the peaks are noticeably broadened indicating more than one species.

Fig. 6 shows the standard curves (peak height *versus* concentration) for monoethylphosphate, diethylphosphate and diethylphosphorothioate, using the conductivity cell response. Excellent linearity over the relatively narrow concentration range was observed. The UV responses for the phosphorothioic acids were linear also and would be expected to remain so over a much broader concentration range. The conductivity response, however, is generally more concentration dependent and would have to be tested before analyzing more concentrated samples.

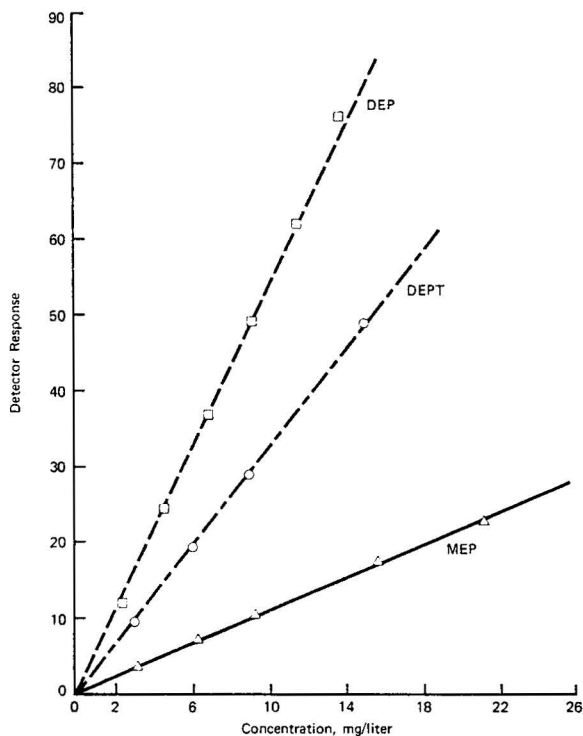


Fig. 6. Standard curves for conductivity detector.

Applications

Organophosphates in waste brines. Organophosphates were determined by the procedure described in this paper in waste brines without any sample preparation. Fig. 7 is a chromatogram of a 250-fold diluted composite brine stream sample from several plants. The conductivity cell response indicates the presence of about 6 ppm DEP and the UV response for DEPT corresponds to 5 ppm in the diluted sample. Improved accuracy of the DEP result could be done using a more dilute eluent if needed.

Hydrolysis rates for diethylphosphorochloridothioate (DEPCT) and dimethylphosphorochloridothioate (DMPCT). The rates of formation of DEPT and chloride

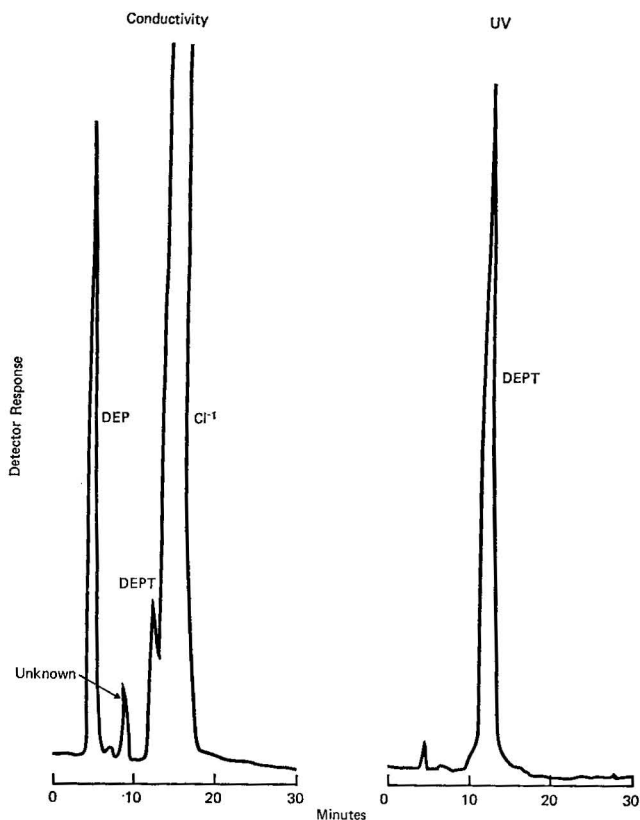


Fig. 7. DEPT in waste brine stream.

ion from DEPCT and DMPT plus chloride from DMPCT were followed using the ion chromatographic system with combined UV and conductivity detection. A 4–6- μ l volume of the compound to be studied was injected into a minimum amount of dry acetonitrile, and the mixture was quickly diluted with water or a weak base to 100 ml. Samples were then periodically withdrawn and analyzed. Fig. 8 shows one set of the chromatograms obtained in this study, and the resulting formation curves for DEPT in water and in the mobile phase are plotted in Fig. 9. As can be seen the compound hydrolyzed very fast in water and faster in the eluent, so the results are not totally accurate. A better technique for this study would be to quench the reaction at about 5-min intervals with a hexane or isooctane extraction to remove the unreacted DEPCT from the water. Then each extracted water sample could be analyzed with no time dependence for the analysis. Nevertheless the study demonstrates the ease for determining the reaction rates and the products formed when phosphorus compounds such as pesticides are hydrolyzed.

Organophosphorus acids in production chloropyrifos. The levels of the organophosphorus acids were determined in production chloropyrifos after they were extracted with deionized water from a carbon tetrachloride solution. A 500 $\times$ 3 mm

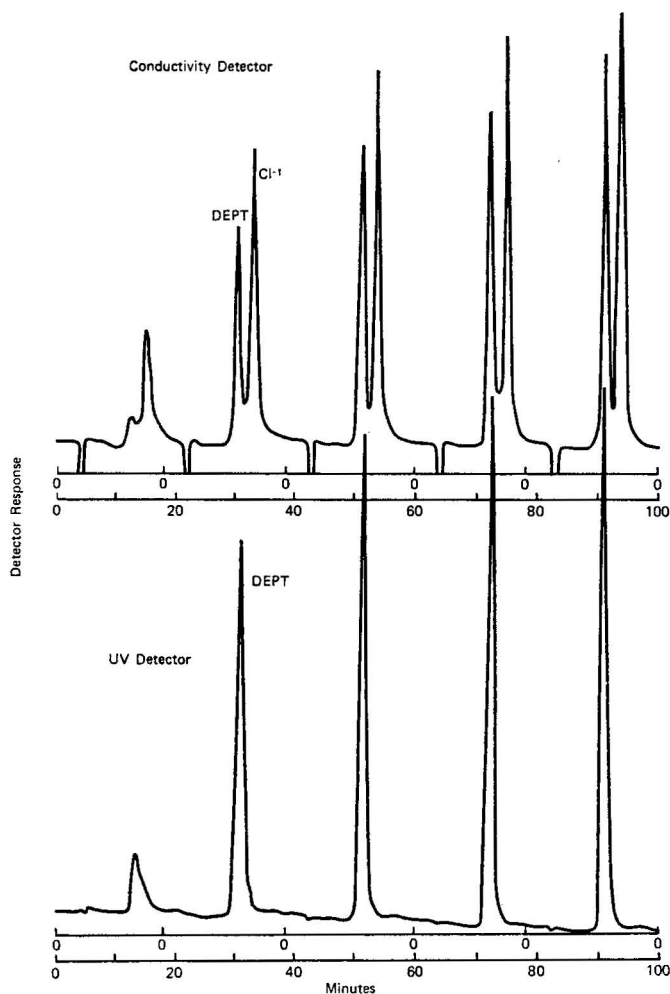


Fig. 8. Chromatograms of DEPCT hydrolysis study.

separating column and a 250×6 mm suppressor column both available from Dionex were used.

Extraction procedure

A 2-g amount of sample was dissolved in 10 ml of carbon tetrachloride and then extracted with 20 ml of deionized water for 5 min on a mechanical shaker. After the phases separated (≈ 1 min), 5 ml of the water layer was filtered into a pre-washed vial using a Swinney filter holder and $5.0 \mu\text{m}$ Mitex (PTFE) Millipore filter. The filtered water was again extracted with 5 ml carbon tetrachloride for 1 min, $50 \mu\text{l}$ of 100-fold concentrated eluent ($0.10 \text{ M Na}_2\text{CO}_3 + 0.1 \text{ M NaHCO}_3$) was added, and then the water phase was removed for subsequent analysis.

Deionized water rather than the eluent was used as extracting solvent to decrease any possible slow hydrolysis of chlorpyrifos which may occur in a basic

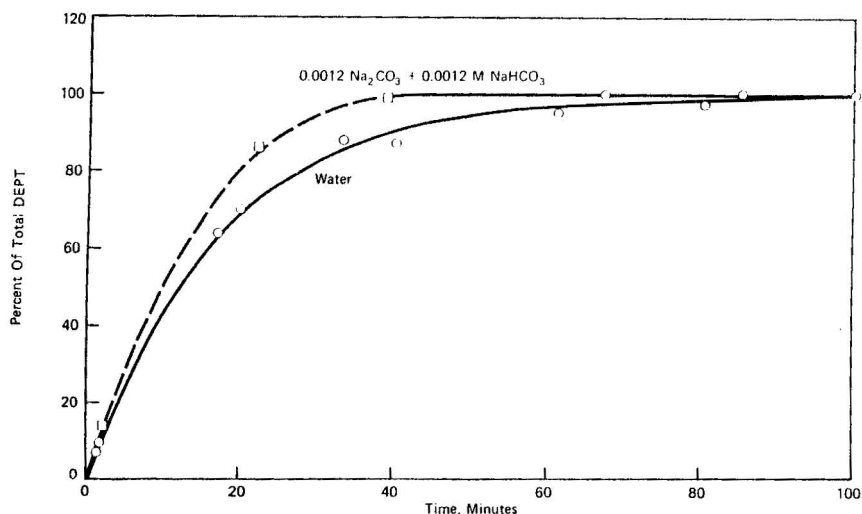


Fig. 9. Hydrolysis of DEPCT to DEPT in water.

solution. The effect of the extraction time on the recovery of methyl phosphorus compounds was evaluated. Known amounts of each compound were added into purified chlorpyrifos-methyl dissolved in carbon tetrachloride. Then 5- and 10-min extractions with deionized water and carbonate-bicarbonate solution were performed. The resulting data listed in Table III indicates very good recoveries using water or the eluent as extracting solvent. However, quantitation of DMP was impossible due to the interference by an unknown. Another unknown detectable with both detectors and which was identified as bromide did not interfere with any of the species of interest. Fig. 10 shows dual detector chromatograms of methyl substituted phosphorus species, phosphoric acid and bromide.

TABLE III
RECOVERY STUDY OF ORGANOPHOSPHORIC ACIDS

Sample	Extraction		DMPT			MMP			PO ₄ ³⁻		
	Solvent	Min	ppm added	ppm found	Recovery (%)	ppm added	ppm found	Recovery (%)	ppm added	ppm found	Recovery (%)
Purified chloro-pyrifos-methyl Eluent	Eluent	5	1.97	1.9	94	1.25	1.2	96	7.16	7.3	102
Purified chloro-pyrifos-methyl Eluent	Eluent	5	3.94	3.9	99	2.50	2.5	100	14.32	14.1	98
Purified chloro-pyrifos-methyl Eluent	Eluent	5	3.94	3.9	99	3.75	3.7	99	21.50	20.5	95
Purified chloro-pyrifos-methyl Water	Water	5	3.44	3.4	99	—	—	—	—	—	—
Purified chloro-pyrifos-methyl Water	Water	10	3.44	3.4	99	—	—	—	—	—	—

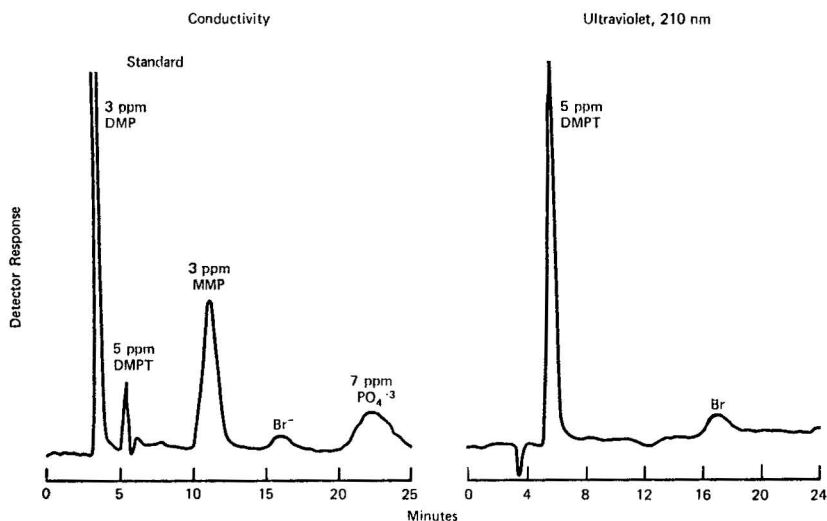


Fig. 10. Dual detector chromatograms of methyl substituted phosphorus species and phosphoric acid.

CONCLUSIONS

The ion chromatographic technique described provides the best truly quantitative procedure for conveniently measuring trace organophosphoric acids in water. Addition of the UV detector to the analytical system permits the simultaneous measurement of UV absorbing phosphorus compounds with the non-absorbing phosphates.

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THIN-LAYER CHROMATOGRAPHY OF 2-METHYL-4-CHLOROPHENOXYACETIC ACID AND ITS SOIL METABOLITES

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SUMMARY

The thin-layer chromatography of 2-methyl-4-chlorophenoxyacetic acid, 4-chloro-*o*-cresol and 3-methyl-5-chlorocatechol and their pentafluorobenzyl derivatives has been studied on silica gel as adsorbent with 19 solvent systems. The best separation of the individual components occurred with toluene-benzene-acetic acid (2:2:1). Chloroform-diethyl ether-toluene (1:1:1) was suitable for the group separation of the pentafluorobenzyl derivatives.

INTRODUCTION

Thin-layer chromatography (TLC) is one of the most widely used techniques for the separation and analysis of pesticides and their residues¹⁻⁸. Walker and Beroza⁹ examined 62 pesticides by TLC with 19 solvent systems on silica gel. TLC has also been recommended for the detection of organochlorine pesticides^{10,11}. Numerous reports describe the application of TLC to separation and clean-up processes in the analysis of chlorinated hydrocarbons¹²⁻¹⁵ and organophosphorus compounds¹⁶⁻¹⁹. Organochloride pesticides have been separated on silica gel^{20,21} and on silica gel-alumina (7:3) layers²². Abbot *et al.*²³ separated herbicide residues by TLC.

In our laboratory, the TLC of chlorinated cresols²⁴ and catechols²⁵ was examined on five different layer materials with different solvent systems in order to develop separation and clean-up methods analyses of these potentially important residues from environmental samples. 2-Methyl-4-chlorophenoxyacetic acid (MCPA) (I) is the most commonly used pesticide in Finland²⁶ and its most important potential residues are its soil metabolites 4-chloro-*o*-cresol (II) and 3-methyl-5-chlorocatechol (III)²⁷. We developed a method for simultaneous determination of I, II and III from soil samples²⁸ in which the crude extract was treated with pentafluorobenzyl bromide in acetone in the presence of potassium carbonate (according to Chau and Terry²⁹) to form derivatives IV, V and VI (see Fig. 1). Then the crude derivative mixture was purified by different methods, including TLC, for final determination by gas-liquid chromatography. In this investigation, the TLC of compounds I-VI on silica gel with different solvent systems has been extended to the separation, identification and determination of these important residues and their derivatives.

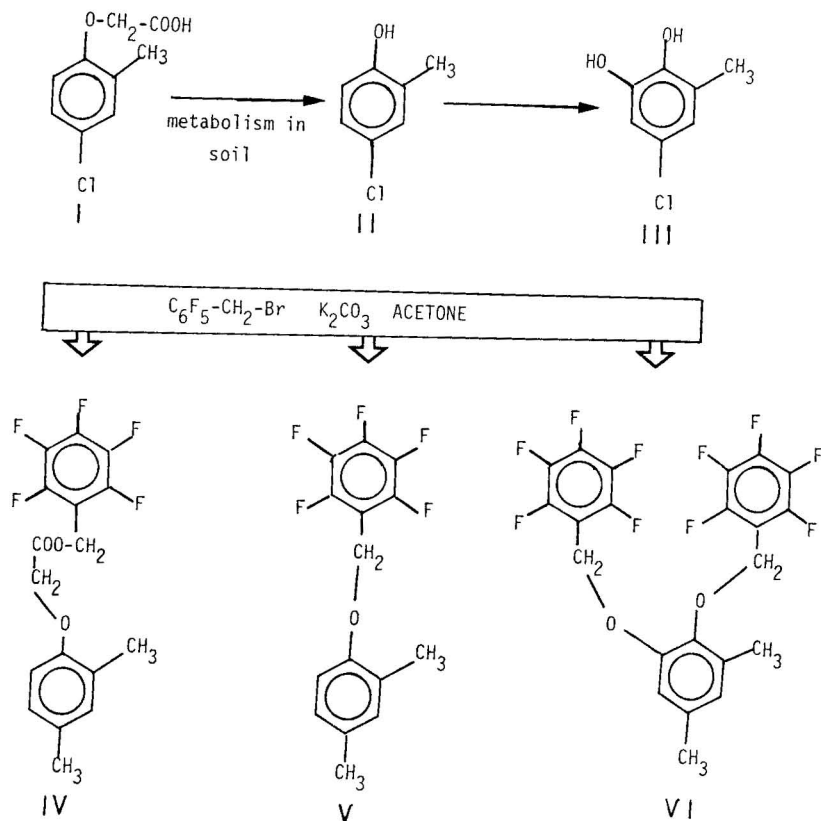


Fig. 1. Structures of MCPA (I), its metabolites in soil, 4-chloro-*o*-cresol (II) and 3-methyl-5-chlorocatechol (III), and their pentafluorobenzyl derivatives (IV-VI).

EXPERIMENTAL

Apparatus and methods

Standard TLC plates (20 × 20 cm; Merck, Darmstadt, G.F.R.) were used. The plates were pre-coated with silica gel G-60 containing a fluorescent indicator, with a layer thickness of 0.25 mm. The samples were spotted with 10–50- μ l pipettes (H. E. Pedersen, Denmark) on a line 1 cm from the bottom of the layer, the first spot being 1 cm from the side of the plate and the following four spots at 4-cm intervals. Ascending elution in a closed glass chamber (Desaga, Heidelberg, G.F.R.) was applied. Both a Desaga scale plate and a metre scale were used to measure the R_F values of the spots.

Samples

MCPA was tried as two different samples: a pure acidic sample was MCPA from Pestanol (99%). MCPA as the sodium salt was a commercial formulation obtained from Kemira (Helsinki, Finland) which contained 4% of 4-chloro-*o*-cresol as the main impurity³⁰. A commercial sample of 4-chloro-*o*-cresol was obtained from Fluka (Buchs, Switzerland) and purified by vacuum distillation. 5-Chloro-3-methylcatechol

was synthesized in our laboratory by J. Knuutinen. The structures of compounds I–III were confirmed with ultraviolet, infrared, nuclear magnetic resonance and mass spectrometry. The purity of the samples was checked by gas chromatography.

Preparation of derivatives IV–VI was performed following the procedure of Chau and Terry²⁹. A 50-mg amount of compound I, II or III was shaken vigorously for 5 min in a glass-stoppered flask with 1 ml of a 1% solution of pentafluorobenzyl bromide in acetone, 500 μ l of a 30% solution of potassium carbonate in water and 4 ml of acetone, and allowed to stand for 3 h at room temperature. The sample was mixed with 2 ml of *n*-hexane and evaporated to dryness with a stream of nitrogen and the residue transferred with diethyl ether into a volumetric flask to make 50 ml of a crude derivative stock solution which represents 1 mg/ml of I, II or III as its derivative IV, V or VI, respectively. Derivatives from MCPA and its sodium salt were identical (IV). The solutions (1 mg/ml) of the original substrates I–III to be applied in the TLC experiments were made up in diethyl ether. The sodium salt of MCPA was insoluble in diethyl ether and therefore was not subjected to TLC.

*Chromogenic reagent*³¹

A 2% solution of 3,5-dichloro-*p*-benzoquinonechlorimine (Merck) in benzene was used for spot detection.

Solvent systems

A preliminary examination of 40 solvents or solvent mixtures was carried out in order to select those giving good spots and reasonable R_F values. All solvent mixtures produced sharp spots. The compositions of the 19 most suitable solvents, all of which except one are mixtures, are listed below (volume ratios):

- (1) dichloromethane–acetone–acetic acid (8:1:1);
- (2) acetone–toluene–acetic acid (2:2:1);
- (3) benzene–ethanol–acetic acid (6:3:1);
- (4) acetone–light petroleum (b.p. 40–60°)–acetic acid (2:2:1);
- (5) acetone–methanol–acetic acid (6:3:1);
- (6) acetone–diethyl ether–acetic acid (2:2:1);
- (7) dichloromethane–toluene–acetic acid (2:2:1);
- (8) acetone–chloroform–acetic acid (2:2:1);
- (9) toluene–benzene–acetic acid (2:2:1);
- (10) dichloromethane–*n*-heptane–acetic acid (2:2:1);
- (11) dichloromethane;
- (12) dichloromethane–acetone–benzene (2:1:1);
- (13) acetone–benzene–acetic acid (5:4:1);
- (14) dichloromethane–*n*-heptane–ethanol (2:2:1);
- (15) chloroform–*n*-hexane–dichloromethane (1:1:1);
- (16) chloroform–diethyl ether–toluene (1:1:1);
- (17) *n*-hexane–*n*-heptane–ethanol (2:2:1);
- (18) dichloromethane–light petroleum (b.p. 40–60°) (2:3);
- (19) toluene–*n*-heptane–diethyl ether (1:1:1).

All solvents for free substrates I–III needed acetic acid to give good results. The derivatives, however, could be tested with other, mainly neutral, solvents (11–19) also.

Development of chromatograms

The development with each solvent system was continued until the solvent front had ascended 17 cm. The plates were then dried in air and sprayed with chromogenic reagent.

RESULTS AND DISCUSSION

All compounds (I–VI) formed sharp spots with all solvent systems used. The spots were yellow with MCPA (I), 4-chloro-*o*-cresol (II) and their pentafluorobenzyl derivatives (IV) and V). The spots from 3-methyl-5-chlorocatechol (III) and its pentafluorobenzyl derivative (VI) were ash grey. The development time, which varied at room temperature from 30 to 90 min, had no marked effect to the spots.

The R_F values obtained with different solvent systems are collected in Table I.

TABLE I

R_F VALUES OF MCPA (I), TWO OF ITS SOIL METABOLITES (II AND III) AND THEIR PENTAFLUOROBENZYL DERIVATIVES (IV–VI) ON A SILICA GEL G-60 LAYER WITH SOLVENT SYSTEMS 1–19

Solvent	Compound						Elution time (min)
	I	II	III	IV	V	VI	
1	0.54	0.82	0.76	0.94	0.83	0.76	60
2	0.56	0.52	0.59	0.71	0.59	0.59	48
3	0.74	0.85	0.79	0.94	0.82	0.82	90
4	0.85	0.88	0.88	0.94	0.91	0.91	90
5	0.79	0.88	0.88	0.91	0.88	0.88	64
6	0.94	0.97	0.90	0.97	0.97	0.97	85
7	0.56	0.59	0.50	0.59	0.59	0.50	45
8	0.88	0.94	0.94	0.94	0.94	0.94	30
9	0.50	0.41	0.56	0.76	0.56	0.44	70
10	0.56	0.59	0.50	0.65	0.62	0.50	55
11				0.62	0.57	0.54	35
12				0.57	0.55	0.57	35
13				0.94	0.88	0.82	50
14				0.62	0.52	0.47	60
15				0.50	0.57	0.52	45
16				0.71	0.73	0.71	50
17				0.41	0.20	0.16	40
18				0.35	0.55	0.41	50
19				0.65	0.41	0.24	47
Colour	Yellow	Yellow	Ash grey	Yellow	Yellow	Ash grey	

The separation in each experiment can be evaluated from these results by comparing the relative differences, x :

$$x = \frac{R_F(\text{I}) - R_F(\text{II})}{R_F(\text{I}) + R_F(\text{II})} \cdot 2 \quad (1)$$

where x is the difference of two R_F values divided by their average. From each three-component TLC experiment, three values of x and their average ($\bar{x}$) are

obtained and the results are given in Table II. It can be concluded that the best separation of the original substrates I–III is achieved with dichloromethane–acetone–acetic (8:1:1) (solvent 1) and toluene–benzene–acetic acid (2:2:1) (solvent 9). The latter is to be preferred because the separation of all three components is greater than $x = 0.1$. Solvent 9 gives a good separation of derivatives IV–VI. However, the acid-free solvents 17, 18 and 19 also give good separations of derivatives IV–VI.

TABLE II

RELATIVE DIFFERENCES (x) BETWEEN R_F VALUES OF I, II AND III OR IV, V AND VI ON SILICA GEL G-60 WITH SOLVENT SYSTEMS 1–19

The value of x is calculated by dividing the difference of two R_F values with their average. Averages of x ($\bar{x}$) for each run are also given.

Solvent	I/II (x)	I/III (x)	II/III (x)	I–III ($\bar{x}$)	IV/V (x)	IV/VI (x)	V/VI (x)	IV–VI ($\bar{x}$)
1	0.412	0.338	0.076	0.275	0.124	0.212	0.088	0.141
2	0.074	0.052	0.126	0.084	0.185	0.185	0.000	0.123
3	0.138	0.065	0.073	0.092	0.136	0.136	0.000	0.091
4	0.035	0.035	0.000	0.023	0.032	0.032	0.000	0.021
5	0.108	0.108	0.000	0.072	0.034	0.034	0.000	0.023
6	0.031	0.043	0.075	0.050	0.000	0.000	0.000	0.000
7	0.052	0.113	0.165	0.110	0.000	0.165	0.165	0.110
8	0.066	0.066	0.000	0.044	0.000	0.000	0.000	0.000
9	0.198	0.113	0.309	0.207	0.303	0.533	0.240	0.359
10	0.052	0.113	0.165	0.110	0.047	0.261	0.214	0.174
11					0.084	0.138	0.054	0.092
12					0.036	0.000	0.036	0.024
13					0.066	0.136	0.071	0.091
14					0.175	0.275	0.101	0.184
15					0.131	0.039	0.092	0.087
16					0.028	0.000	0.028	0.019
17					0.689	0.877	0.222	0.596
18					0.444	0.158	0.292	0.298
19					0.453	0.921	0.523	0.632

For the use of TLC as a clean-up stage prior to the gas chromatographic determination of MCPA and its metabolites^{28,32,33}, small values of x and $\bar{x}$ are required, which allows to take a narrow zone of the layer for the purified group extract. On the other hand, acetic acid is an unsuitable component of the solvent as it contaminates the sample and thus affects electron-capture detection. As the gas chromatography of compounds I–III is difficult and the sensitivity of the electron-capture detector towards them is low, only the TLC of derivatives IV–VI with acid-free solvents (11, 13–19) can be considered in the clean-up. Tables I and II indicate that chloroform–diethyl ether–toluene (1:1:1) (solvent 16) is the best, and consequently it is recommended for the group separation (clean-up) of the pentafluorobenzyl derivatives IV–VI of MCPA and its metabolites. In our previous analyses, dichloromethane (solvent 11) was used²⁸ successfully, which is confirmed by the present results through the low x values (Table II).

CONCLUSIONS

The herbicide MCPA can be analysed together with its metabolites 4-chloro-*o*-cresol and 3-methyl-5-chlorocatechol by TLC on silica gel with developing solvents containing acetic acid, or as their pentafluorobenzyl derivatives with neutral organic solvent mixtures. The best overall separation of all components is achieved with toluene-benzene-acetic acid (2:2:1). For group separation of the pentafluorobenzyl derivatives, chloroform-diethyl ether-toluene (1:1:1) is recommended.

ACKNOWLEDGEMENT

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CACTUS ALKALOIDS

XL. IDENTIFICATION OF MESCALINE AND OTHER β -PHENETHYL-AMINES IN *PERESKIA*, *PERESKIOPSIS* AND *ISLAYA* BY USE OF FLUORESCAMINE CONJUGATES

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SUMMARY

The combined methods of analytical thin-layer chromatography (TLC), high-performance TLC, and gas-liquid chromatography (GLC) have been used to identify specific alkaloids in twelve cactus species. TLC of fluorescamine conjugates, prepared from primary amines in the alkaloid extracts, served to substantiate results from the other chromatographic identifications. Traces of the hallucinogen, mescaline, were identified and quantitated by GLC in *Islaya minor* Backbg., *Pereskia corrugata* Cutak, *Pereskia tampicana* Weber, and *Pereskiopsis scandens* Br. and R., demonstrating that this alkaloid is distributed through all three cactus tribes.

INTRODUCTION

Fluorescamine, 4-phenylspiro[furan-2(3H),1'-phthalan]-3,3'dione (Fluram; Roche, Nutley, N.J., U.S.A.), was first synthesized and utilized by Weigele and co-workers¹⁻³ for the fluorimetric assay of amino acids and primary amines. It has been used as a chromatographic spray reagent to detect amino acids^{4,5}, peptides⁶ and cactus alkaloids (both primary and secondary amines)⁷. Imai *et al.*⁸ have detected amino acids, peptides and other primary amines by first conjugating these compounds with fluorescamine and then performing analytical thin-layer chromatography (TLC). Farid⁹ has recently used high-performance liquid chromatography to assay the fluorescamine conjugate of aminocaproic acid.

Our previous isolation and identification of many alkaloids in the cactus tribes *Opuntiae* and *Cereeae* served as an impetus for phytochemical examination of the previously uninvestigated third cactus tribe, the *Pereskiae*. The problem of obtaining suitable quantities of these rare, leafy, tropical species for alkaloid isolations prompted our development of a simple microanalytical technique, employing TLC separation of fluorescamine conjugates, to substantiate results obtained by conventional TLC and gas-liquid chromatography (GLC) of the non-derivatized alkaloids.

EXPERIMENTAL

Plant material

Cuttings of the cacti (Table I) were obtained from authenticated plants growing at Grigsby Cactus Gardens, Vista, Calif., U.S.A.; Abbey Gardens, Carpinteria, Calif., U.S.A.; Fairchild Tropical Gardens, Miami, Fla., U.S.A.; Botanical Gardens, The University of Michigan, Ann Arbor, Mich., U.S.A.; and the Desert Botanical Gardens, Phoenix, Ariz., U.S.A. Reference photographs are on file. Specimens were frozen, freeze-dried and passed through a 2-mm screen in a Wiley mill. Samples weighing 20 g, or proportionally less, were extracted, as previously described¹⁰, to yield extracts A (alkaloids), B (non-alkaloidal material) and C (water soluble alkaloids). Extracts A and C were pooled for each species and subjected to anion-exchange chromatography¹¹, to separate the phenolic and non-phenolic alkaloids.

Analytical TLC

Analytical TLC and, in some cases, radial high-performance TLC (HPTLC)¹² were utilized to determine the presence of alkaloids in the phenolic and non-phenolic alkaloid extracts. For analytical TLC, silica gel plates (Merck 60, F-254, 5 × 20 cm) were spotted with 5–15 μ l of the extracts (in 1.0 ml of ethanol) or with reference alkaloids (1–5 μ g) and chromatographed in solvent systems A, B, E, F and G as previously described¹³. A Camag 28600 Series U-chamber system with a digital solvent delivery system (flow-rate set at 1 μ l/sec) was utilized for HPTLC¹², employing the same five solvent systems on special silica gel plates (Merck 60, F-254, for nano-TLC, 5 × 5 cm). Evidence for the presence of a specific alkaloid in the extracts was based on co-chromatography with the reference alkaloid in the five different solvent systems. Visualization was accomplished by spraying sequentially with fluorescamine, dansyl chloride and tetrazotized benzidine or iodoplatinate^{7,11}. Fluorescamine conjugates were separated by TLC on the silica gel plates using ethyl ether–glacial acetic acid (19:1) and visualized under long wave UV light; for HPTLC of the conjugates ethyl acetate–methanol (17:2) and ethyl ether–ethanol (2:1) were also found to be useful.

Gas-liquid chromatography

Traces of mescaline (3,4,5-trimethoxy- β -phenethylamine) and 3,4-dimethoxy- β -phenethylamine were detected by TLC and HPTLC in extracts of several of the cactus species screened (Table I). GLC on a Varian Series 2700 gas chromatograph equipped with a flame-ionization detector and employing a 1.5 m × 0.32 cm 1.5% OV-101 (stainless steel) column (chromosorb G, 100–200 mesh) was performed on the non-phenolic alkaloid fractions to quantitate the amount of mescaline (Table II). The oven temperature was set at 150°, and the nitrogen carrier gas flow was 30 ml/min. Concentrations of alkaloids were estimated by comparing peak areas of reference standards to those from the non-phenolic fractions. Nearly identical retention times and peak height enhancement with references served to substantiate the alkaloid identities in the extracts.

Preparation of fluorescamine conjugates

Fluorescamine conjugates of eleven β -phenethylamine (β -PEA) alkaloid reference standards (mescaline; 3,4-dimethoxy- β -PEA; 2,5-dimethoxy- β -PEA; β -hydroxy-

TABLE I

ALKALOID SCREENING RESULTS AND METHODS OF ANALYSIS

Species screened and source*	Alkaloid identified	Method of analysis			
		TLC**	TLC of fluorescamine conjugate	HPTLC***	GLC***
<i>Islaya minor</i> Backbg. (T.) from Grigsby	3,4-Dimethoxy- β -phenethylamine	×	×	×	×
	Mescaline	×	×	×	×
	β -Phenethylamine	×	×		
	Corypalline	×			
	Hordenine	×			
	3-Methoxytyramine	×	×		
	N-Methyltyramine	×			
	Tyramine	×	×		
<i>Melocactus delessertianus</i> Lem. from Abbey	Pellotine	×			
	Tyramine	×		×	
<i>M. maxonii</i> (Rose) Gürke from Abbey	Tyramine	×		×	
<i>Pereskia aculeata</i> Miller from Fairchild	Tyramine	×	×		
<i>P. autumnalis</i> (Eichlam) Rose from Univ. of Michigan	β -Phenethylamine	×	×		
	Tyramine	×	×		
<i>P. corrugata</i> Cutak from Fairchild	3,4-Dimethoxy- β -phenethylamine	×	×	×	×
	Mescaline	×	×	×	×
	3-Methoxytyramine	×	×		
	Tyramine	×	×		
<i>P. cubensis</i> Br. and R. from Univ. of Michigan	Tyramine	×	×		
<i>P. godseffiana</i> (Sand.) Knuth from Univ. of Michigan	Tyramine	×	×		
<i>P. grandifolia</i> Haw. from Univ. of Michigan	<i>p</i> -Methoxy- β -hydroxy- β -phenethylamine	×	×		
	3-Methoxytyramine	×	×		
	Tyramine	×	×		
<i>P. grandiflora</i> Hort. from Univ. of Michigan and Desert Bot. Gard.	β -Hydroxymescaline	×	×		
	Tyramine	×	×		
<i>P. pititache</i> (Karwinsky) Br. and R. from Abbey	β -Phenethylamine	×	×		
	Tyramine	×	×		
<i>P. tampicana</i> Web. from Univ. of Michigan	3,4-Dimethoxy- β -phenethylamine	×	×	×	×
	Mescaline	×	×	×	×
	<i>p</i> -Methoxy- β -hydroxy- β -phenethylamine	×	×	×	
	β -Phenethylamine	×	×		
	Tyramine	×	×		

(Continued on p. 82)

TABLE I (continued)

Species screened and source*	Alkaloid identified	Method of analysis			
		TLC**	TLC of fluorescamine conjugate	HPTLC***	GLC***
<i>Pereskopsis chapistle</i> (Web.) Br. and R. from Abbey	<i>p</i> -Methoxy- β -hydroxy- β -phenethylamine	×	×		
	β -Phenethylamine	×	×		
	3-Methoxytyramine	×	×		
	Tyramine	×	×		
<i>P. scandens</i> Br. and R. from Abbey	3,4-Dimethoxy- β -phenethylamine	×	×	×	×
	Mescaline	×	×	×	×
	Tyramine	×	×		

* For source explanation see *Plant material* section.

** Co-chromatography with reference materials in five systems using solvents and visualization aids as previously described^{7,13}.

*** HPTLC and GLC were used primarily to provide additional evidence for the identifications of 3,4-dimethoxy- β -phenethylamine and mescaline.

TABLE II

GLC DATA AND MESCALINE QUANTITATION

3,4 dm PEA = 3,4-Dimethoxy- β -phenethylamine. All injection volumes 10 μ l; ref = reference alkaloid; np = non-phenolic alkaloid extract.

Plant/dry weight of sample	Peak*	Retention time (sec)	Calculated amount alkaloid injected (μ g)	Total mescaline in sample (μ g)	Mescaline in plant (%)
<i>I. minor</i> (20 g)	Mescaline (ref)	337	20	—	—
	3,4 dm PEA (ref)	200	20	—	—
	Mescaline (np)	337	17	340	0.0017
	3,4 dm PEA (np)	200	38	760	0.0038
<i>P. corrugata</i> (20 g)	Mescaline (ref)	400	30	—	—
	3,4 dm PEA (ref)	200	30	—	—
	Mescaline (np)	395	5	100	0.0005
	3,4 dm PEA (np)	197	9	180	0.0009
<i>P. tampicana</i> (12 g)	Mescaline (ref)	337	30	—	—
	3,4 dm PEA (ref)	195	30	—	—
	Mescaline (np)	330	8	160	0.0013
	3,4 dm PEA (np)	190	15	300	0.0025
<i>P. scandens</i> (20 g)	Mescaline (ref)	400	10	—	—
	3,4 dm PEA (ref)	216	10	—	—
	Mescaline (np)	395	2.2	44	0.0022
	3,4 dm PEA (np)	211	2.9	58	0.0029

mescaline; 3,5-dihydroxy-4-methoxyamphetamine; 3-methoxy-4-hydroxy- β -PEA; normetanephrine; 4-methoxy- β -PEA; β -PEA; 4-methoxy- β -hydroxy- β -PEA; and tyramine) were prepared by dissolving 1 mg of primary amine hydrochloride in 0.4 ml of distilled water and 2.5 mg of sodium carbonate; 3 mg of fluorescamine was dissolved in 0.2 ml of acetone and added to the primary amine alkaloid solution.

The reaction mixture was immediately shaken on a Vortex mixer for 15 sec, and 1.0 ml of distilled water followed by 1.0 ml of ethyl acetate was added. The aqueous layer was extracted, and two additional 1.0 ml portions of ethyl acetate were used for subsequent extractions. The combined ethyl acetate extracts were reduced to a small volume under rotary vacuum and analyzed immediately by TLC and HPTLC. Although secondary amines produce UV quenching when sprayed with fluorescamine⁷, no conjugates were detected by using the above procedure with five different secondary amines (such as N-methylmescaline and N-methyltyramine). Samples of several alkaloid-fluorescamine conjugates were prepared in the above way and submitted for mass spectrometry (MS) analyses to confirm the expected molecular weights (Table III).

TABLE III

MASS SPECTRUM AND TLC RESULTS OF ALKALOID-FLUORESCAMINE CONJUGATES

Fluorescamine conjugate	R_F^* in ethyl ether-glacial acetic acid (19:1)	Spectrum type	Observed parent ion (m/e)	Calculated MW	
				Acid conjugate	Lactone
2,5-Dimethoxy- β -phenethylamine	0.68	—	—	—	—
3,4-Dimethoxy- β -phenethylamine	0.44	CI-MS	442	459	441
β -Hydroxymescaline	0.35	—	—	—	—
3-Methoxy-4-hydroxy- β -phenethylamine	0.51	—	—	—	—
4-Methoxy- β -phenethylamine	0.69	CI-MS	412	429	411
Mescaline	0.41	EI-MS	411	489	471
		CI-MS	472		
		EI-MS	471		
β -Phenethylamine	0.73	CI-MS	382	399	381
		CI-MS	382		
		EI-MS	381		
Tyramine	0.60	—	—	—	—

* Average of four determinations.

A larger 270- μ mol portion of fluorescamine was reacted with an equivalent amount of mescaline hydrochloride to prepare enough mescaline conjugate for closer characterization (Fig. 1). Upon volume reduction of the ethyl acetate, yellow crystals of the conjugate were obtained; however, these required reversed-phase low-pressure chromatography on a 0.6×30 cm column of C_{18} -silica gel (methanol-water-acetonitrile, 5:3:1) for purification: m.p. 120–124° (from ethyl acetate); 54% yield; UV $\lambda_{\text{max}}^{\text{methanol}}$ (ϵ) 275 nm (18,200) and 390 nm (6450); IR $\nu_{\text{max}}^{\text{KBr}}$ 3300–2500 cm^{-1} (broad),

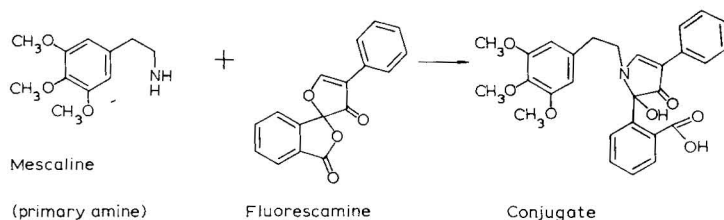


Fig. 1. The fluorescamine conjugate of mescaline.

1680 cm^{-1} (sh), and 1230 (sh); chemical ionization mass spectrometry (CI-MS) m/e 472 ($M+H^+$ of the lactone); electron impact (EI)-MS exact mass 471.167 (calc. 471.168 for the lactone) and low resolution m/e 181 ($M-190$ from β -cleavage of mescaline); ^1H nuclear magnetic resonance (NMR) (80 MHz, CDCl_3) δ 2.78 (m, 2H, $\beta\text{-CH}_2$ of mescaline), δ 3.21–3.41 (m, 2H, $\alpha\text{-CH}_2$ of mescaline), δ 3.52, 3.70 and 3.75 (bs, s, s, 3 mescaline ring CH_3O 's), δ 6.23 (bs, 2H, o -protons of mescaline), the remainder of the spectrum consisted of overlapping and ill-defined peaks integrating to account for the remaining ten conjugated and aromatic protons.

The conjugate preparation procedure was extended to the phenolic and non-phenolic cactus alkaloid fractions. The alkaloid extract was condensed to dryness and 0.5–1.0 ml of 1 *N* sodium carbonate was added; the remainder of the preparation was identical to that described above for the reference alkaloids with the exception that five, rather than three, ethyl acetate extractions were made. The conjugated extracts were then subjected to TLC (Table I).

RESULTS AND DISCUSSION

Chromatographic results of the alkaloid screenings for many of the cactus species investigated indicated the presence of a number of phenolic and non-phenolic alkaloids. The primary amines identified by conventional TLC and HPTLC had these identifications confirmed by TLC of their fluorescamine conjugates. Table I lists the alkaloids identified in each species and the methods of analysis used. In each case the TLC identification of primary amines in the extracts was confirmed by TLC of their fluorescamine conjugates; HPTLC and GLC were used primarily to provide additional evidence for the identifications of 3,4-dimethoxy- β -phenethylamine and mescaline. Table II summarizes the GLC data and includes estimates of mescaline concentrations.

Mass spectral data for the fluorescamine conjugates of representative alkaloids confirmed that the conjugates are converted to their respective lactones when heated¹, e.g., in the MS probe. Table III shows the MS results and R_F values of the alkaloid-fluorescamine conjugates.

These results demonstrate a new method for identifying small quantities (100–500 pg, refs. 5, 7) of primary β -phenethylamines. The specificity of fluorescamine for primary amines makes it superior to dansyl chloride which will also form conjugates with secondary amines, imidazoles and phenols. The method should be useful for alkaloid identifications in cases where large quantities of plant material cannot be obtained for actual alkaloid isolation and where an additional method is needed to substantiate TLC and GLC results that employ the underivatized alkaloids. A disadvantage of the method is the relative instability of the fluorescent conjugates which are both heat and acid labile^{1,14}. Conjugates chromatographed in the ethyl ether–acetic acid (19:1) system lost fluorescence within one hour after development.

Tyramine was found in the phenolic fraction of all the cactus species screened; thus, the presence of this alkaloid seems to be a common feature of the *Pereskia* and their relatives. Since the other β -phenethylamines likely involve tyramine as a biogenetic precursor¹⁵, the widespread occurrence of tyramine is not surprising. The detections of β -phenethylamine, corypalline, β -hydroxymescaline and 4-methoxy- β -hydroxy- β -phenethylamine are either novel or unusual in the cactus family, and

isolation studies are needed to confirm these chromatographic identifications.

Traces of mescaline and 3,4-dimethoxy- β -phenethylamine were identified in *I. minor*, *Pereskia corrugata*, *P. tampicana* and *Pereskiopsis scandens*. An individual would have to ingest kilograms of these plant materials to receive a hallucinogenic dose of mescaline, so these species pose no drug abuse problems. However, the discovery of this alkaloid in these cacti is still interesting. Its presence in *Pereskiopsis* (tribe *Opuntieae*), *Islaya* (tribe *Cereeae*) and *Pereskia* (tribe *Pereskieae*) indicates that the genetic ability to produce mescaline is common to all three tribes of the cactus family. The capacity to accumulate large quantities of mescaline, however, has yet to be demonstrated among the *Opuntieae* and the *Pereskieae*^{16,17}.

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CHROM. 12,433

Note

Gas chromatography-mass spectrometry of the flophemesyl derivatives of a series of fatty acid methyl ester chlorohydrins

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The formation and assessment of pentafluorophenyldimethylsilyl ether (shortened to flophemesyl) derivatives for the gas chromatographic (GC) analysis of steroids was first reported by Morgan and Poole^{1,2}, who subsequently extended their work and showed the sensitivity obtainable for analysis by electron-capture detection of flophemesyl derivatives of alcohols, phenols, amines and carboxylic acids^{3,4}. They also demonstrated that flophemesyl ethers have good mass spectral (MS) properties showing some marked differences to the trimethylsilyl (TMS) ethers of steroids⁵. Our own investigations of methyl ester chlorohydrins have previously led us to report the GC-MS analysis of the TMS derivatives⁶, but in practice we have subsequently found that for trace analysis of chlorohydrins in polymer systems⁷ the flophemesyl derivatives are a better alternative, enabling sensitive electron-capture detection and producing simple but very characteristic MS fragmentation patterns. In this note we report the ease of formation, and stability of the flophemesyl derivatives of a series of chlorohydrin fatty acid methyl esters, their electron-capture limits of detection and MS fragmentation patterns.

EXPERIMENTAL

The preparation of the fatty acid ester chlorohydrins was previously described⁶, and the flophemesyl derivatives were formed quantitatively by addition of equal volumes of pyridine and flophemesylamine (Lancaster Synthesis, St. Leonard Gate, Lancaster, Great Britain) allowing to stand at room temperature for a few minutes, for complete reaction.

GC was performed on a Pye Series 104 equipped with flame ionisation and ⁶³Ni electron-capture detectors. The glass column (1.5 m × 0.6 cm O.D.) packed with 3% OV-1 on Diatomite CLQ (100-120 mesh) was operated at 260° with a nitrogen carrier gas flow-rate of 60 ml/min. The electron-capture detector was operated at 300° in a pulsed mode with a spacing of 150 μsec.

Combined GC-MS was carried out on a DuPont Model 21-490B mass spectrometer interfaced with an all glass jet separator to a Varian 2700 gas chromatograph. The separator and connecting lines were held at 270°, the ion source at 230° and mass spectra were obtained at 70 eV scanning over the mass range from *m/e* 20 — 800 at 4 sec per decade.

RESULTS AND DISCUSSION

Flophemesyl derivatives of fatty acid ester chlorohydrins exhibited good GC properties with sharp peaks, an absence of tailing and no evidence of decomposition. Solutions of the derivatised chlorohydrins were stable at room temperature in diethyl ether solvent for up to 72 h without evidence of any hydrolysis to the parent compound. The electron-capture detector was approximately 10^3 times more sensitive than the flame ionisation detector to equal amounts of flophemesyl chlorohydrins with a practical working limit of detection by electron capture of the order of 5 pg.

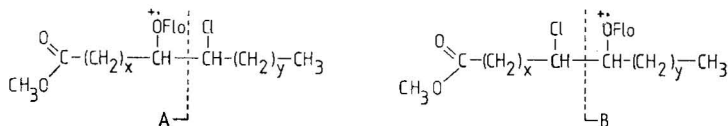
TABLE I

CHARACTERISTIC IONS FROM FLOPHEMESYL CHLOROHYDRINS

Spectra were obtained by combined GC-MS.

Starting material	Fragmentation ions			
	A		B	
	<i>m/e</i>	Relative intensity (%)	<i>m/e</i>	Relative intensity (%)
Methyl 9,10-epoxyoctadecanoate	411	100	367	71
Methyl 9,10-epoxyhexadecanoate	411	100	339	84
Methyl 6,7-epoxyoctadecanoate	369	100	409	82
Methyl 11,12-epoxyoctadecanoate	439	100	339	36
Methyl 11,12-epoxyeicosanoate	439	100	367	77
Ethyl 9,10-epoxyoctadecanoate	425	100	367	50

The mass spectra of the flophemesyl chlorohydrins showed few small fragment ions but in each case produced two intense characteristic ions the (two major ions in each spectra). These ions A and B (shown in Table I) were due to cleavage between the adjacent carbons bearing the chlorine and O-flophemesyl groups.



Analogously to the TMS chlorohydrins⁶ the formation of positional chlorohydrin isomers by reaction of hydrogen chloride with epoxides is again illustrated by this fragmentation pattern. The spectra are characterised by (i) an absence of other ions of major intensity, (ii) the absence of molecular ions—in contrast to the MS behaviour of simple flophemesyl alcohols³, (iii) the $M - 15$ ion being absent—or occasionally present at very low intensity, (iv) the $M - 31$ ion commonly being the highest ion present (intensity commonly between 3 and 8%). A typical mass spectrum of the flophemesyl derivative of the reaction product of HCl with methyl 9,10-epoxyoctadecanoate is illustrated in Fig. 1.

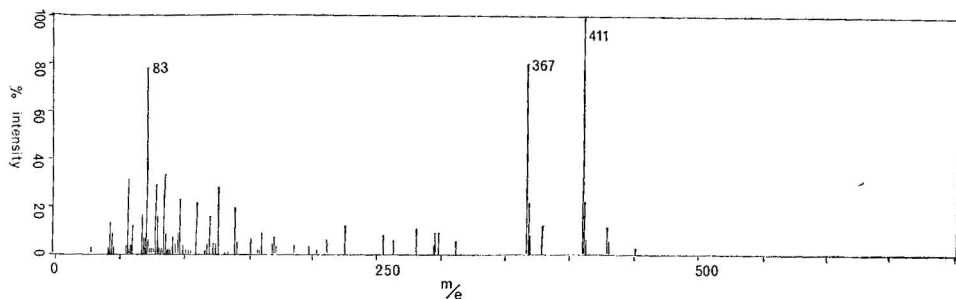


Fig. 1. Mass spectrum of the flophemesyl derivative of the reaction product of methyl 9,10-epoxyoctadecanoate with HCl.

In these laboratories flophemesyl derivatives have been routinely employed for the electron-capture capillary GC analysis of chlorohydrins isolated from polymer systems⁷ and have proved well suited for quantitation at high sensitivity.

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Note

Gas chromatographic determination of Lenacil and Pyrazon in soil and bacterial cultures

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The fate of herbicides in soil has been widely studied. Pyramin and venzar are commonly used as weed control agents for sugar beet and in horticulture^{1,2}. Sensitive methods are required for determining trace amounts of Lenacil and Pyrazon in soil and bacterial cultures in order to provide reliable information about their behaviour under different conditions.

Spectrophotometric and thin-layer and gas chromatographic (GC) methods^{3–5} have been described for the analysis of Lenacil and Pyrazon residues. Pease⁴ developed a method for determining Lenacil by GC, but interpretation of the chromatograms was difficult and long retention times were obtained. No GC method for the determination of both Lenacil and Pyrazon has been reported.

The technique described here is based on the extraction of Lenacil and Pyrazon with an organic solvent, drying with magnesium sulphate, evaporation at 40 ° and GC determination using a flame-ionization detector (FID).

EXPERIMENTAL

Apparatus and reagents

A Model 18.3.6 gas chromatograph (Veb Chromatron, Berlin, G.D.R.) with a FID was equipped with a stainless-steel column (100 cm × 3 mm I.D.) packed with 3% SE-30 plus 6% OV-210 on Gas-Chrom Q (80–100 mesh) (Applied Science Labs., State College, Pa., U.S.A.). A Type 350 rotatory evaporator (Unipan, Warsaw, Poland) was used.

Venzar (commercial product containing 80% of Lenacil) and analytical-grade standard Lenacil (99.8% purity) were obtained from DuPont (Geneva, Switzerland). Pyramin (commercial product containing 80% of Pyrazon) and analytical-grade standard Pyrazon (99.8% purity) were obtained from BASF (Ludwigshaven, G.F.R.).

Chloroform, ethyl acetate and anhydrous magnesium sulphate (analytical-reagent grade) were obtained from POCh (Gliwice, Poland).

Materials

Loess soil from Biały Kościół at a depth of 5–15 cm ($C_{org} = 1.24\%$, $N = 0.12\%$, $pH_{H_2O} = 7.2$) was used. A control soil, soil containing 100 ppm of venzar, soil containing 200 ppm of pyramin and soil containing 100 ppm of venzar plus 200 ppm pyramin were prepared. All samples were enriched with 5 ml/kg of a microflora suspension from fresh garden soil and were incubated at room temperature. The water capacity of the soil was maintained at 30% (w/w).

Soil samples for analysis were taken after 4 h and 6 months of incubation; 25 g of dry soil sample were placed in a Soxhlet apparatus and extracted with chloroform for 2 h. The extracts obtained were dried with 5 g of anhydrous magnesium sulphate and evaporated to dryness at 40°.

Azotobacter vinelandii, strain A66, was cultivated in Burk's medium prepared according to Dalton and Postgate⁶. Doses of herbicides half those in the experiments with soil were introduced into the media. Medium without herbicides constituted the control sample. Herbicides were autoclaved separately in distilled water and added under sterile conditions. A 1-ml volume of 48-h-old pre-cultures of strain A66 was used as an inoculum. The experiments were carried out at 28° on a rotary shaker (60 rpm).

Post-culture media for analysis were taken after 1 h and 9 days of incubation. A 50-ml volume of sample was introduced into a separating funnel and, after adjustment of the pH to 9.0, were extracted three times with 50-ml portions of chloroform and three times with 50-ml portions of ethyl acetate. The extracts were combined, dried with 5 g of anhydrous magnesium sulphate and evaporated to dryness. All samples were examined triplicate.

Calibration graphs

Calibration graphs were prepared from standard solutions containing 0.35–5 μg of venzar and 1–10 μg of pyramin per microlitre of chloroform (see Fig. 1).

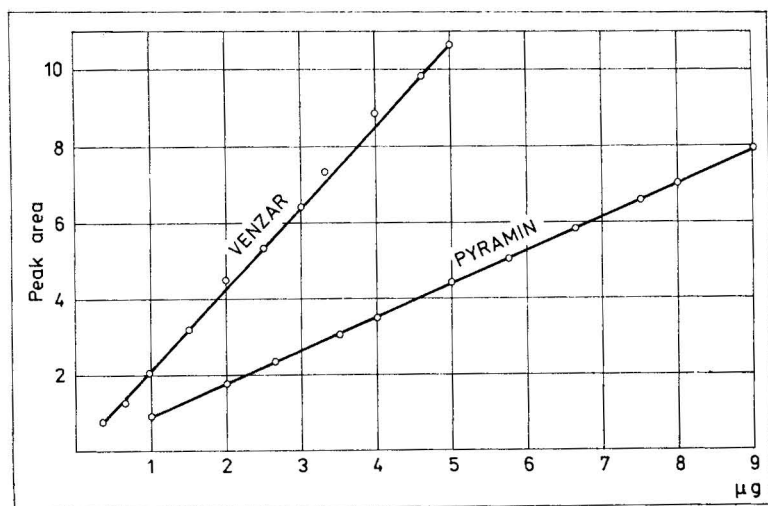


Fig. 1. Calibration graphs for pyramin and venzar.

Chromatography

The residues of the herbicides were dissolved in 1 or 2 ml of chloroform. Standard solutions and residue solutions were chromatographed using the column specified above with injector, column and detector temperatures of 295°, 230° and 310°, respectively, and nitrogen as the carrier gas at a flow-rate of 80 ml/min. Samples of 1 μ l were injected. The amounts of the herbicides present were calculated from the calibration graphs.

TABLE I

GLC ANALYSIS OF SOIL TREATED WITH HERBICIDES

Time of incubation	Herbicide	Sample			
		Control	With pyramin (200 ppm)	With venzar (100 ppm)	With pyramin (200 ppm) and venzar (100 ppm)
4 h	Pyramin	0.0	168.0	0.0	158.0
	Venzar	0.0	0.0	96.4	96.2
6 months	Pyramin	0.0	30.5	0.0	34.5
	Venzar	0.0	0.0	32.6	70.3

TABLE II

GLC ANALYSIS OF LIQUID MEDIA OF *AZOTOBACTER VINELANDII* STRAIN A66 AFTER INCUBATION

Time of incubation	Herbicide	Sample			
		Control	With pyramin (100 ppm)	With venzar (50 ppm)	With pyramin (100 ppm) and venzar (50 ppm)
1 h	Pyramin	0.0	96.3	0.0	94.9
	Venzar	0.0	0.0	43.5	44.5
9 days	Pyramin	0.0	94.1	0.0	92.0
	Venzar	0.0	0.0	42.2	43.1

RESULTS AND DISCUSSION

Results of studies with pyramin and venzar added to untreated soil and bacterial media after incubation are shown in Tables I and II. Recoveries obtained with this method (on the basis of the calibration graphs, Fig. 1) from soil averaged 81.0% and 96.3%, and from liquid media 95.6% and 86.0% of pyramin and venzar, respectively. The amounts of herbicides after incubation for 6 months in soil and 9 days in media with *Azotobacter vinelandii* are also given. A typical gas-liquid chromatogram of standard solutions of the investigated herbicides is shown in Fig. 2. Chromatograms of herbicides extracted from enriched samples of soil and liquid media are shown in Figs. 3 and 4. The detection limits of the method are 0.016 ppm and 0.08 ppm of venzar and pyramin, respectively, based on a 25-g sample. The retention times of Lenacil and Pyrazon were 460 and 615 sec, respectively.

The method described was very useful for the simultaneous determination of pyramin and venzar residues. In comparison with retention times and chromatograms in the literature⁴, our technique is characterized by simplicity of the chromatograms,

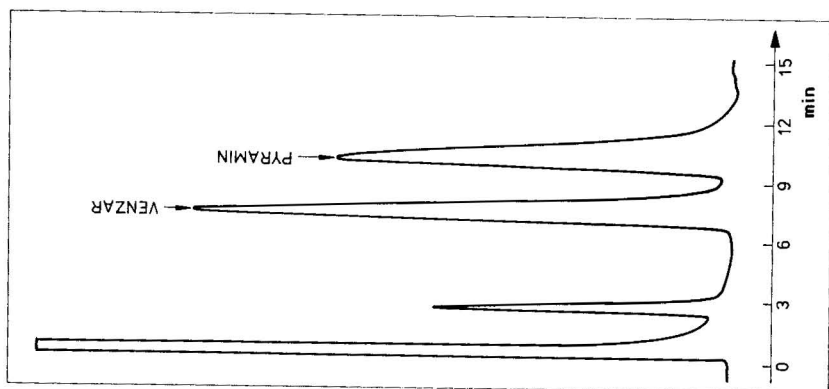
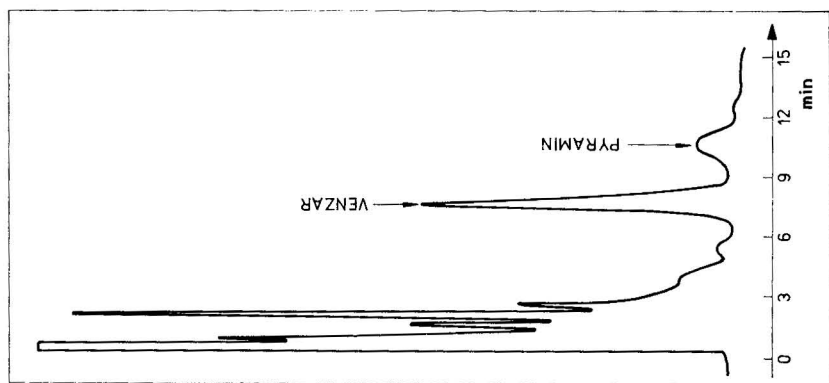
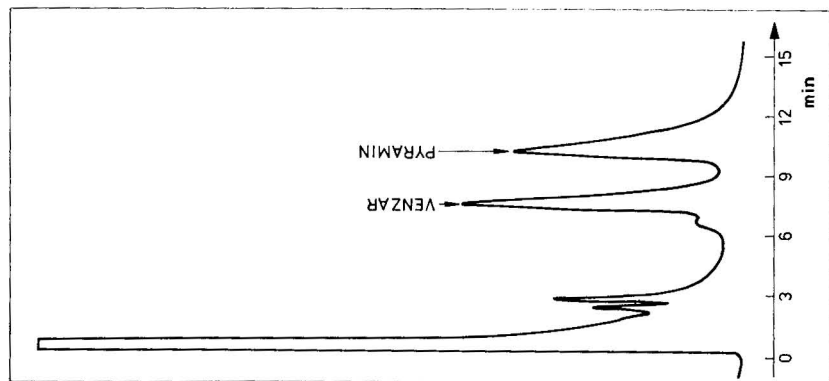


Fig. 2. Gas-liquid chromatogram of standard solution of pyramin and venzar.

Fig. 3. Gas-liquid chromatogram of pyramin and venzar extracted from soil enriched with 200 and 100 ppm of the respective herbicides after incubation for 6 months.

Fig. 4. Gas-liquid chromatogram of pyramin and venzar extracted from bacterial medium enriched with 100 and 50 ppm of the respective herbicides after incubation for 9 days.

a shorter experimental time and a two-times higher sensitivity. The proposed system is suitable for routine operation. However, for other biological materials (plant tissues), it is necessary to develop a technique of purifying the sample material. These aspects are being studied.

ACKNOWLEDGEMENTS

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Note

Screening procedure for therapeutic benzodiazepines by high-performance liquid chromatography of their benzophenones

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Benzodiazepines can be analysed either as the compounds themselves and their metabolites, or as their hydrolysis products, *i.e.*, benzophenones.

The analysis of benzodiazepines and their metabolites is well known, but most studies have been focused on one or a few compounds. The most commonly used technique is gas-liquid chromatography, preferably with an electron-capture detector, as the molecules involved are electronegative¹⁻¹². High-performance liquid chromatography (HPLC) has been used for the same purpose¹³⁻¹⁸. These methods are specific and sensitive but, because of their complexity (extraction at specific pH, derivatization, variable chromatographic conditions), they are not easily applicable in screening tests.

On the other hand, benzodiazepines are often analysed as benzophenones after acid hydrolysis. With biological materials the sensitivity of detection is increased after hydrolysis as a benzodiazepine and its corresponding metabolites yield the same benzophenone(s). Separation of the benzophenones is performed by thin-layer chromatography (TLC) and they are detected under UV light or with Bratton-Marshall reagent if a primary amine is present¹⁹⁻²¹. Quantification is not possible, however.

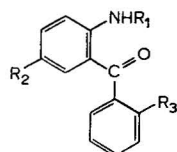
HPLC is the method of choice for screening benzodiazepines as their benzophenones: the separation is more reliable than with TLC and the benzophenones are simultaneously quantified. The analysis consists simply of acid hydrolysis of the sample, followed by a single injection of the solution after addition of an internal standard. Identification and quantification are performed by calculating relative retention times and relative peak heights.

EXPERIMENTAL

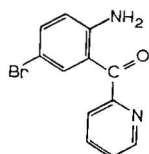
Materials

The apparatus consisted of a Hewlett-Packard liquid chromatograph with a built-in fixed-wavelength UV detector (254 nm). Hibar column pre-packed with LiChrosorb RP-8 of particle diameter of 7 μm was used. This was selected after examining RP-3, RP-8 and RP-18 columns; a particle diameter of 7 μm gave better results than 10 μm . The benzophenones listed in Table I were obtained from La Roche (Basle, Switzerland), except for ADB, which was synthesized¹⁸. Camazepam was provided by Sintesa (Milan, Italy).

TABLE I
BENZOPHENONES EXAMINED



R_1	R_2	R_3	Abbreviation	Systematic name
CH ₃	Cl	H	MACB	5-Chloro-2-(methylamino)benzophenone
H	Cl	H	ACB	2-Amino-5-chlorobenzophenone
H	Cl	Cl	ADB	2-Amino-5-chloro-2'-chlorobenzophenone
CH ₃	NO ₂	F	MANFB	2'-Fluoro-2-(methylamino)-5-nitrobenzophenone
H	NO ₂	F	ANFB	2-Amino-2'-fluoro-5-nitrobenzophenone
H	NO ₂	H	ANB	2-Amino-5-nitrobenzophenone
NH ₂	NO ₂	Cl	ANCB	2-Amino-2'-chloro-5-nitrobenzophenone



ABBP

2-(2-Amino-5-bromobenzoyl)pyridine

Chromatographic conditions

The mobile phase was methanol-water (1:1) and the column pressure was kept at 350 bar with a solvent flow-rate of 3.02 ml/min. A six-port valve loop injector with a 10- μ l loop was used. The recorder speed was 38 cm/h. All work was carried out at ambient temperature.

Standard mixture

A standard mixture was prepared from the eight benzophenones listed in Table I (derived from the hydrolysis products of the thirteen 1,4-benzodiazepines

TABLE II
BENZODIAZEPINES AND THEIR BENZOPHENONE HYDROLYSIS PRODUCTS

Benzodiazepine	Benzophenone*
Bromazepam (Lexotan)	ABBP
Camazepam (Albego, Paxor)	MACB, ACB
Chlordiazepoxide (Librium)	ACB
Di-K-Chlorazepate (Belseren, Tranxene)	ACB
Demethyldiazepam (Madar)	ACB
Diazepam (Valium)	MACB, ACB
Lorazepam (Temesta)	ADB
Medazepam (Nobrium)	MACB, ACB
Oxazepam (Seresta)	ACB
Temazepam (Levanxol)	MACB, ACB
Flunitrazepam (Rohypnol)	MANFB, ANFB
Nitrazepam (Mogadon)	ANB
Clonazepam (Rivotril)	ANCB

* For abbreviations, see Table I.

listed in Table II), each at a concentration of 50 $\mu\text{g/ml}$ in methanol. Camazepam was added as the internal standard at the same concentration.

RESULTS

Fig. 1 shows a chromatogram, obtained under the above conditions, of the standard mixture containing the eight benzophenones and the internal standard. Retention times relative to camazepam (with their standard deviations) were calculated. The absolute retention time of camazepam is about 11 min 20 sec.

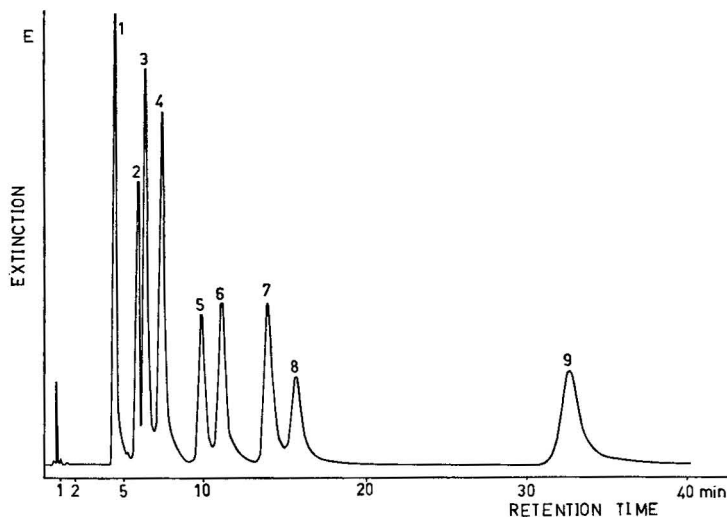


Fig. 1. Chromatogram of the standard mixture. Peaks: 1 = ABBP; 2 = ANFB; 3 = ANB; 4 = ANCB; 5 = MANFB; 6 = camazepam (internal standard); 7 = ACB; 8 = ADB; 9 = MACB.

The peak-height ratio of benzophenone to internal standard was used for quantification. Relative peak heights were calculated (with their standard deviations).

The results are given in Table III. For quantitative work a standard deviation of less than 2.5% for each of the benzophenones is obtained.

TABLE III

RELATIVE RETENTION TIMES (RR_t) AND RELATIVE PEAK HEIGHTS (R_h) WITH THEIR STANDARD DEVIATIONS (σ)

Compound	RR_t	$\sigma_{RR_t} (n = 31)$	R_h	$\sigma_{R_h} (n = 16)$
ABBP	0.399	0.004	2.725	0.032
ANFB	0.532	0.004	1.694	0.033
ANB	0.573	0.006	2.380	0.042
ANCB	0.668	0.006	2.088	0.044
MANFB	0.893	0.008	0.923	0.009
Camazepam	1		1	
ACB	1.265	0.009	0.975	0.024
ADB	1.421	0.010	0.533	0.012
MACB	2.958	0.027	0.566	0.012

Linearity of the detector response was tested for ABBP and camazepam (0.1–1000 $\mu\text{g/ml}$). The correlation coefficient of the linear regression between absorption and concentration was 0.994 for both products. Quantification over the whole concentration range was possible.

CONCLUSION

Eight benzophenones of thirteen 1,4-benzodiazepines could be separated and identified with the use of an internal standard. The products could be quantified by means of their relative peak heights with a standard deviation of less than 2.5%. Although the identification of a compound is the first requirement in toxicology, quantification is of particular interest for monitoring purposes, as the benzodiazepines are used not only as tranquillizers and hypnotics but also as anticonvulsants. The proposed screening method identifies and quantifies them simply and rapidly.

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Note

Analysis of cyclodextrins by high-performance liquid chromatography

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The chromatographic separation of cyclodextrins has already been described¹⁻⁵ and a high-performance method for the rapid chromatographic analysis of mixtures of α -, β - and γ -cyclodextrins (α -CD, β -CD and γ -CD, consisting of six, seven and eight α -1,4-linked glucose units, respectively) has been published recently⁶.

A method using a column packed with a silica derivative containing amino groups, with acetonitrile-water mixtures as the eluent, has been described for the analysis of mixtures of mono- and disaccharides^{7,8}. However, this method does not seem very suitable for the direct analysis of the formation of cyclodextrins from starch, as the low solubility of starch in the eluent may cause problems during the chromatographic run.

The chromatographic analysis of linear oligosaccharides, produced from starch by enzymatic or chemical degradation, is mostly carried out on cation-exchange resins at elevated temperature with water as the eluent⁹. We have found this method to be suitable for the separation and analysis of cyclodextrins.

EXPERIMENTAL

The cyclodextrins (α -CD, β -CD and γ -CD) were obtained from Sigma (St. Louis, Mo., U.S.A.).

Standard solutions of cyclodextrins or linear oligosaccharides at concentrations up to 20 mg/ml were prepared.

A Hewlett-Packard Model 1084B liquid chromatograph, equipped with an automatic sampling system and a Model HP 1031 differential refractometer as a detector, was used. The column system consisted of two columns (25×0.62 cm I.D.), connected by unions of low dead volume. Both columns were packed with Aminex 50W-X4 (Ca^{2+}) (20-30 μm). The temperatures of the columns and the detection system were 90° and 37°, respectively. Only water, filtered through 0.2- μm filters and deaerated at 90°, was used for elution.

RESULTS

Using the standard conditions for the analysis of linear oligosaccharides, it was found that α -CD, β -CD and γ -CD were much more retarded on the column

system than their linear analogues. Some results are shown in Fig. 1. Clearly the interactions of the cyclodextrins with the cation-exchange resin are different from those of the linear analogues, which are assumed to be based on complex formation between the calcium ions of the resin and the hydroxyl groups of the glucose molecules^{10,11}.

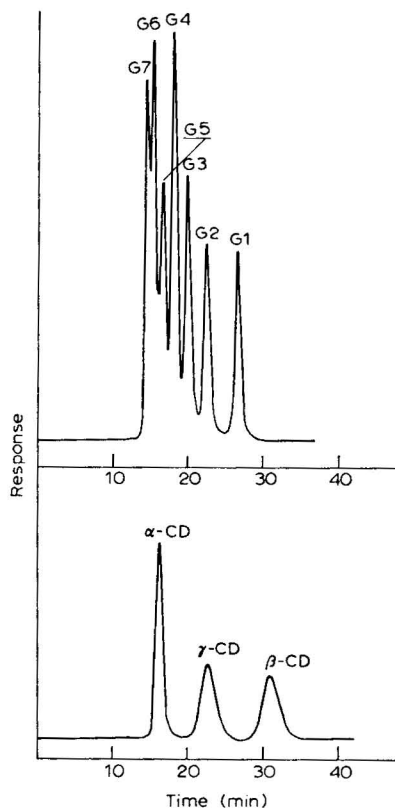


Fig. 1. Chromatographic separation of linear gluco-oligosaccharides (G_1 – G_7) and cyclodextrins on a cation-exchange resin. Columns (two in series), 25×0.62 cm I.D., 90° ; eluent, water, 90° ; flow-rate, 0.3 ml/min; resin, Aminex 50W-X4 (Ca^{2+}) (20 – $30 \mu\text{m}$); detection, differential refraction at 37° ; sample size, $50 \mu\text{l}$; recorder chart speed, 0.2 cm/min.

At lower column temperatures the retention times of β -CD and γ -CD increased (Fig. 2) and the peaks broadened considerably. With the linear glucose oligomers no such change in retention times was observed. With α -CD only a small but consistent increase in retention time was found.

Cyclodextrins form inclusion complexes with many low-molecular-weight organic and inorganic compounds¹², and recently, it has been reported that they also show interactions with polymers in aqueous solution, *e.g.*, a polystyrenesulphonic acid solution¹³. The long retention times of the cyclodextrins might reasonably be explained by such interactions with the polystyrene backbone of the resin.

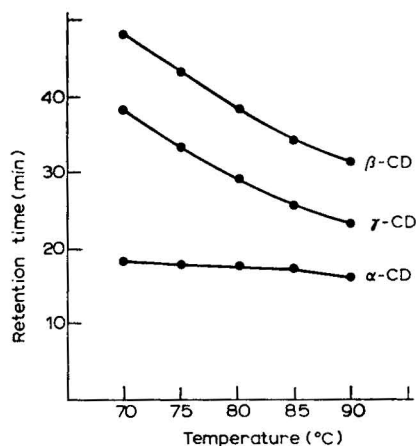


Fig. 2. Retention times of cyclodextrins at different column temperatures. Other conditions as in Fig. 1.

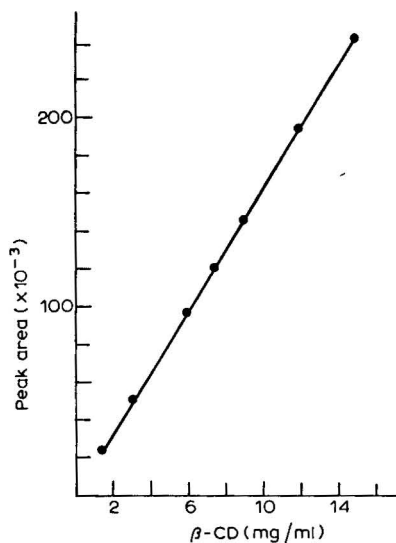


Fig. 3. Relationship between peak area and β -CD concentration. Conditions as in Fig. 1; detector attenuation, 4.

The chromatographic system described here provides a method for the analysis of cyclodextrins in the presence of many other gluco-oligosaccharides. The considerable temperature dependence of the retention times of β -CD and γ -CD makes the system flexible. With appropriate calibration the method can be used quantitatively, as shown in Fig. 3 for β -CD by the relationship between peak area and cyclodextrin concentration.

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CHROM. 12,422

Note

Separation of rat thyroid lysosome subpopulations on Sepharose 2B

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Although gel chromatography is widely used for molecular fractionation, it has rarely been used for subcellular fractionation. This paper describes an attempt to separate a crude thyroid lysosomal population into subpopulations, characterized by the criteria of age and size, using Sepharose 2B gel.

Thyroid lysosomes are involved in the degradation of thyroglobulin (Tg), an iodinated glycoprotein that is the source of thyroid hormones. The intralysosomal hydrolysis of Tg is known to be heterogeneous, as recently iodinated (new) Tg is hydrolysed faster than the previously iodinated (old) Tg^{1,2}; thus, one can consider that, taking the iodine specific radioactivity (SRA = new label/old label) as an index, the higher the SRA the younger is the lysosome. The size distribution of organelles was studied by morphometry. A relationship between the size distribution of the lysosomes and the mean "age" of their iodine content has been found and could be interpreted as separation of the lysosomes into physiological subpopulations.

MATERIALS AND METHODS

Biological material

Male Wistar rats, weighing about 250–300 g and housed at $23 \pm 1^\circ$, received 50 μ g of iodine daily. They were maintained in isotopic equilibrium with ¹²⁵I as described by Simon³. Two hours before killing they received one intraperitoneal injection of 250 μ Ci of carrier-free ¹³¹I in 0.5 ml of 0.9% sodium chloride solution. Thyroid lobes were homogenized in a Tris·HCl (0.1 M)–saccharose (0.15 M) medium of pH 7.0, and the bulk of iodinated particles was prepared as described by Simon *et al.*⁴. Briefly, a 34,000 g lysosomal pellet was obtained by re-suspending the washed pellet in the above elution medium.

Gel chromatography

Sixty grams of Sepharose 2B (Pharmacia, Uppsala, Sweden) were washed three times by gentle agitation with distilled water in order to discard the azide. After re-suspension in the elution medium, the gel was poured into a glass column of I.D. 2.5 cm. All of the experiments were performed at 4°.

In some experiments, the void volume (V_0) of the column was determined before chromatography by measuring the elution volume of a 2% solution of Blue Dextran 2000 (Pharmacia).

The total population of lysosomes (2-ml sample) of at least four pooled thyroids was applied on to the column and eluted with about 100 ml of the elution medium. Fractions of 20 drops were collected with a Gilson fraction collector. The complete organic iodine recovery was obtained only after a second elution with the elution medium containing 1% Triton X-100.

Measurement of radioactivity

The ^{131}I and ^{125}I activities of each fraction collected were measured using a two-channel Packard Autogamma spectrometer. The ^{127}I content of each fraction was calculated from its ^{125}I activity as described by Simon³, and the distribution of old (^{127}I) and new (^{131}I) iodine was followed with respect to their specific radioactivity (SRA = parts per 1000 of ^{131}I injected per microgram of ^{127}I).

Morphometric study

Aliquots of the first subpopulation of lysosomes eluted in the void volume of the column (L_I) and the total population (L_T) were centrifuged for 1 h at 80,000 g. Each pellet was immersed at 4° for 1 h in 2 ml of 2% glutaraldehyde solution in 0.1 M phosphate buffer (pH 3), post-fixed in 1% osmium tetroxide solution and embedded in Araldite. Sections 300–600 Å thick were prepared with a LKB microtome and stained with 2% potassium permanganate solution.

The morphology of the pellet was observed with a Hitachi HU 12 electron microscope. The morphometric study was carried out on photographs taken at the final magnification of 33,000 from each field selected at random. Each photograph was superimposed at random with a network of parallel lines, and the number and the length of intersections between these lines and organelle sections were recorded. For each population, results from about 1000 measurements were totalled and used for the calculation of its representative distribution of organelle diameters. The method of calculation will be published elsewhere⁵.

RESULTS

The lower part of Fig. 1 shows the results of thyroid lysosome chromatography on a sample from four rats in isotopic equilibrium. The ^{125}I label permits the determination the iodine pool within each particulate subpopulation: (1) the first subpopulation (L_I) was eluted in the void volume (V_0) and represents $17.67 \pm 4.43\%$ ($n = 9$); (2) the second subpopulation (L_{II}) was eluted at about $2V_0$ and represents $15.17 \pm 13.54\%$ ($n = 5$); (3) the third subpopulation (L_{III}) is strongly bound to the gel, its iodinated content being discharged only after solubilization of lysosomal membranes with Triton X-100, and represents $46.31 \pm 2.72\%$ ($n = 5$). The fractions between the peaks represent about 20% and complete recovery of iodine ($99.0 \pm 1.1\%$) ($n = 5$) is therefore obtained.

The recovery of L_I is independent of the sample size as the mean recovery is always about 18% ($18.21 \pm 2.43\%$) ($n = 5$) for samples obtained from 4, 7, 28 and 36 rats.

A comparison of the iodine SRA of each subpopulation, as shown in the upper part of Fig. 1, demonstrates that $\text{SRA } (L_I) > \text{SRA } (L_{II}) > \text{SRA } (L_{III})$. This result is obtained after a single injection of ^{131}I 2 h before killing.

Fig. 2 shows the frequency of true organelle diameters calculated from the

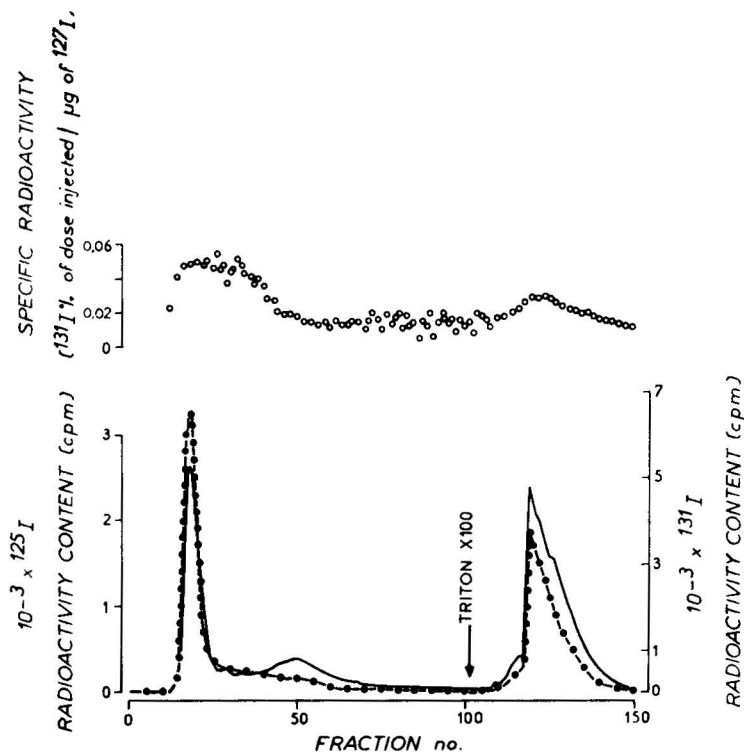


Fig. 1. Fractionation of a total population of thyroid lysosomes with Sepharose 2B. Four rats, isotopically equilibrated with ^{125}I , were injected with $250\ \mu\text{Ci}$ of ^{131}I 2 h before killing. The total population of lysosomes was layered on the chromatographic column equilibrated with $0.15\ M$ sucrose buffered with $0.1\ M$ Tris-HCl (pH 7). Elution was carried out successively with the same medium (100 ml) and the same medium containing 1% of Triton X-100 (500 ml). In the lower diagram, the ^{125}I (—) and ^{131}I (●—●) profiles show the successive elution of three subpopulations of lysosomes, L_I , L_{III} and L_{II} . The ^{125}I content of each fraction was calculated, then its specific radioactivity (SRA) was calculated with respect to ^{131}I . The upper diagram shows that the L_I subpopulation contains the higher proportion of newly iodinated molecules.

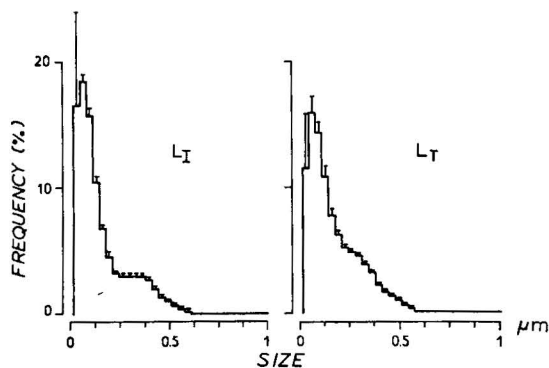


Fig. 2. Morphometry of the total population and subpopulation L_I of rat thyroid lysosomes. Each histogram gives the frequency of each size class of organelles calculated from observed sizes. The bars indicate standard errors for the classes. The ranges of true diameters are similar but the profiles are different.

observed diameters of the total population L_T and the subpopulation L_I . Both populations have a true diameter in the range 0.03–0.6 μm range. The maximum frequency for both L_T and L_I is observed at an organelle diameter between 0.03 and 0.07 μm , but there are 20% less organelles in the range 0.25–0.40 μm for L_I compared with L_T . This is statistically significant at the level $P < 0.05$ (t -test).

DISCUSSION

A crude thyroid lysosomal population can be separated into three main subpopulations by Sepharose 2B chromatography. The delayed elution of L_{III} and the adsorption of L_{II} could be a function of the size of the organelles. Systematic studies by Gerlich *et al.*⁶ of the elution of particles of known sizes on Sepharose 2B have shown that (1) K_{av} diminishes as their size increases and (2) only organelles of maximum diameter 0.08 μm were eluted with total recovery, and those of diameter over 0.08 μm were adsorbed to the gel; 80% of 1- μm diameter organelles were eluted compared with only 50% of 0.3- μm organelles. Their results indicate that only the smallest organelles (maximum diameter 0.1 μm) are entirely represented in L_I and that L_{III} consists mainly of the largest organelles. Our results also suggest that L_{II} is composed of vesicles with diameters in the range 0.2–0.5 μm . Hence Sepharose 2B chromatography would roughly separate lysosomes into three subpopulations with $L_I < L_{II} < L_{III}$ in terms of their size. This is confirmed, at least in part, by our morphometric study in which we have pointed out that if L_I is composed of all organelle sizes it is significantly low in 0.25–0.40- μm organelles.

Moreover, the proposed method permits the separation of lysosome subpopulations according to their "age". Thus, taking the SRA as an index, the youngest lysosomes are the smallest and the oldest are the largest, as $\text{SRA}(L_I) > \text{SRA}(L_{II}) > \text{SRA}(L_{III})$. As we have previously suggested⁷, it can be proposed that successive fusion of lysosomes between themselves leads to their progressive maturation, as revealed by the size increase that is associated with a decrease in content of young iodinated molecules. The biological implication of this interpretation will be published elsewhere.

In conclusion, Sepharose 2B chromatography is a useful method for the fractionation of the thyroid lysosomal subpopulations. It is interesting that, for a given gel volume, column size and sample volume, and with the concentration of the sample loaded on to the column varying by a factor of 9, the recovery of lysosomal iodine was complete and the recovery of each subpopulation was neither drastically nor systematically modified. Therefore, this method is highly reproducible and the origin of the fluctuation seems to be biological rather than technical. Moreover, although at least one subpopulation (L_{II}) can never be recovered as integral particles, it should be emphasized that the use of a pure lysosomal sample will permit comparative studies between thyroid lysosomal subpopulation contents: soluble molecules (iodinated molecules, enzymes) or solubilized molecules derived from membranes.

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Note

Presence of sodium azide during acid hydrolysis of protein samples causes the destruction of tyrosine, phenylalanine and histidine

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We have observed that certain protein samples presented to us for amino acid analysis did not appear to contain the amino acids tyrosine, phenylalanine and histidine, although the samples were expected to contain these amino acids. We have shown that destruction of these amino acids has been occurring during sample hydrolysis in 6 *N* HCl due to the presence of small amounts of sodium azide which was present (as an antibacterial agent) in the phosphate buffered saline in which the proteins were originally presented.

MATERIALS AND METHODS

The chromosomal protein HMG2 (mol. wt. 26,000) which has been extensively studied and well characterised in our laboratories in recent years^{1,2} was used as our test protein in the following experiment. Protein was dissolved in water containing 0.1 % (w/v) sodium azide, at a protein concentration of 1 mg/ml. A 50- μ l aliquot was taken and dried down under vacuum in a Pyrex tube. 6 *N* HCl (0.5 ml) was added, the tube sealed, and the sample hydrolysed at 110° for 24 h. The concentration of sodium azide in the hydrolysing acid was, therefore, 0.01 % (w/v). The protein hydrolysate was then dried under vacuum and amino acid analysis carried out using a Rank-Hilger Chromaspek amino acid analyser. An identical control experiment was carried out but in the absence of sodium azide.

RESULTS AND DISCUSSION

Table I presents the results of the amino acid analyses, where sample hydrolysis was carried out both in the presence (analysis 1) and absence (analysis 2) of sodium azide. The most obvious features are the complete loss of tyrosine and phenylalanine in the azide containing sample (analysis 2). Additionally, histidine is almost completely destroyed and the peak due to the methionine sulphone has disappeared. An extremely high ammonia peak also prevented the resolution of arginine in this analysis. Additional unknown peaks were observed in the second analysis. Two unknown peaks eluted between histidine and lysine. These are presumably degradation products from the destroyed amino acids. The only other difference between the 2 analyses is an

TABLE I

AMINO ACID ANALYSIS OF TEST PROTEIN IN THE PRESENCE AND ABSENCE OF SODIUM AZIDE

<i>Amino acid</i>	<i>Recovery (nmoles)</i>	
	<i>Analysis 1 (6 N HCl)</i>	<i>Analysis 2 (6 N HCl + sodium azide)</i>
Mes	13.4	—
Asp	72.9	81.7
Thr	16.9	16.9
Ser	61.1	58.2
Glu	138.5	136.6
Pro	50.0	49.4
Gly	53.3	51.6
Ala	61.3	58.8
Cys	—	—
Val	15.1	14.1
Gln	12.7	12.2
Leu	15.7	14.9
Tyr	21.1	—
Phe	30.0	—
His	14.4	2.7
Lys	146.4	144.5
Arg	34.1	*
NH ₃	60.0	>10 ³

* Not resolved from large ammonia peak.

increase in the aspartic acid peak by about 15% in analysis 2, which suggests that a further degradation product is running in this position.

The amount of sodium azide present in our hydrolysing medium (0.01 % w/v), is, of course, extremely small, but even lesser amounts are likely to cause at least partial destruction of some amino acids. We have shown with previous samples that dialysing the protein againsts distilled water overnight is sufficient to remove the sodium azide, resulting in a normal amino acid analyses.

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Letter to the Editor

A hazard in high-performance liquid chromatography

Modern techniques of high-performance liquid chromatography may prove hazardous unless due care is given to proper handling of solvents. Some of the hazards involved are less obvious than others, such as their possible toxicity. The handling of pressurized liquids and sources of liquid under pressure must be especially cautious. We tend to think that liquids are soft and ignore the strength of a liquid jet. The following accident illustrates this kind of hazard.

Recently, one of my friends was trying to unpack a silica column. After taking off the metal frit which closed it, he connected the pump and started to pump isooctane at a high flow-rate and waited. Nothing happened, and the solvent was percolating gently through the column. Trying to scratch some silica out with a small wire, the chemist triggered the rapid expulsion of all of the silica and received a strong jet of silica and solvent on the thumb and the side of the hand. It was a slightly painful slap, he said, but he just washed his hand and nobody else in the laboratory noticed anything.

In the evening the hand was painful and the chemist could not sleep. In the morning his thumb and hand were swollen so he went to hospital, where he was given a pain killer and an appointment with a specialist for the following day. By then the hand was very painful, very swollen and dark. Surgery was decided, first to cut off the necrosed tissues, then to graft skin over the wound.

After 2 weeks of hospital care my friend is back in his laboratory, happy and healthy and more cautious.

It seems that the penetration of a considerable amount of isooctane, a non-toxic solvent, under the effect of the shock and not the shock itself, was responsible for the necrosis of skin tissues. Think what could happen with toxic solvents such as chloroform, acetonitrile or methanol, or with the benzene-carbon tetrachloride mixtures used for column packing! In addition to the local skin necrosis, general poisoning could occur.

Chromatographers should therefore take precautions against possible jets of solvents under high pressure.

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Liquid Column Chromatography

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10. CARBOHYDRATES

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19. PROTEINS

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19. PROTEINS

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20g. Lyases

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- 1712 Drescher, D.G.: Purification of blood carbonic anhydrases and specific detection of carbonic anhydrase isoenzymes on polyacrylamide gels with 5-dimethylaminonaphthalene-1-sulfonamide (DNSA). *Anal. Biochem.*, 90 (1978) 349-358 - polyacrylamide gel.
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- 1717 Mencher, D. and Reshef, L.: Effect of triamcinolone on renal and hepatic phosphoenolpyruvate carboxykinase in the newborn rat. Changes in the rate of synthesis of the enzyme and in the activity of its translatable messenger RNA. *Eur. J. Biochem.*, 94 (1979) 581-589 - SDS-polyacrylamide gel.
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- 1720 McGuire, J.J. and Rabinowitz, J.C.: Studies on the mechanism of formyltetrahydrofolate synthetase. The *Peptococcus aerogenes* enzyme. *J. Biol. Chem.*, 253 (1978) 1079-1085 - polyacrylamide gel.
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- 1722 Panasenko, S.M., Alazard, R.J. and Lehman, I.R.: A simple, three-step procedure for the large scale purification of DNA ligase from a hybrid λ lysogen constructed *in vitro*. *J. Biol. Chem.*, 253 (1978) 4590-4592 - polyacrylamide gel.

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- 1729 Venner, H., Hartmann, M. and Walter, F.: (Modern separation processes for nucleic acids). *Ergeb. Exp. Med.*, 32 (1979) 41-45; *C.A.*, 90 (1979) 99321y.

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- 1732 Zeeberg, B. and Caplow, M.: Reactions of tubulin-associated guanine nucleotides. *J. Biol. Chem.*, 253 (1978) 1984-1990 - polyacrylamide gel.

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- 1733 Alonso, A., Flytzanis, C.N., Schätzle, U., Scheller, K. and Sekeris, C.E.: Purification and reverse transcription of the messenger RNA coding for the insect protein, calliphorin, isolated from larvae of the blowfly, *Calliphora vicina* R.-D. *Eur. J. Biochem.*, 94 (1979) 601-608 - polyacrylamide gel.
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- 1741 Doenecke, D.: Modification of DNA in chromatin with methyltransferase from *Haemophilus influenzae* Rd. *Eur. J. Biochem.*, 93 (1979) 481-486 - polyacrylamide gel.
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- 1762 Bald, E.: Analytical utility of 2-halopyridinium salts. I. Paper electrophoretic characterization of thiols as 2-alkyl(aryl)thio-1-methylpyridinium-p-toluenesulphanates. *J. Chromatogr.*, 174 (1979) 483-487 - paper.

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- 1763 Armbrrecht, H.-J., Wasserman, R.H. and Bruns, M.E.H.: Effect of 1,25-dihydroxy-vitamin D₃ on intestinal calcium absorption in strontium-fed rats. *Arch. Biochem. Biophys.*, 192 (1979) 466-473 - immunoelectrophoresis, polyacrylamide gel.
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30b. Chloroplast and other natural pigments

- 1765 Henriques, F. and Park, R.B.: Characterization of three new chlorophyll-protein complexes. *Biochem. Biophys. Res. Commun.*, 81 (1978) 1113-1118 - polyacrylamide gel.
- 1766 Markwell, J.P., Thornber, J.P. and Boggs, R.T.: Higher plant chloroplasts: Evidence that all the chlorophyll exists as chlorophyll-protein complexes. *Proc. Natl. Acad. Sci. U.S.A.*, 76 (1979) 1233-1235; *C.A.*, 90 (1979) 164969g.
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- 1773 Sato, K.: (Diagnosis of hypergammaglobulinemia by thin-layer isoelectric point electrophoresis). *Kitazato Igaku*, 8 (1978) 338-340; *C.A.*, 90 (1979) 83095s.

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33b. Anions

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35. SOME TECHNICAL PRODUCTS AND COMPLEX MIXTURES

35d. Complex mixtures and non-identified compounds

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- 1787 Tweto, J., Hou, E. and Doyle, D.: Purification and characterization of an externally accessible 55,000-molecular weight protein of hepatoma tissue culture cells. *Arch. Biochem. Biophys.*, 193 (1979) 422-430 - SDS-polyacrylamide gel, isoelectric focusing.
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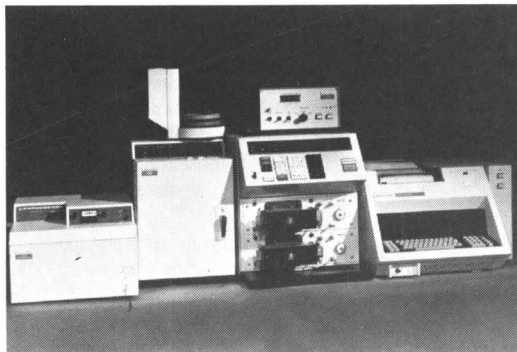
journal of chromatography news section

APPARATUS

N-1333

AUTOMATED METHODS PROCESSOR FOR HPLC

Perkin-Elmer announces the availability of an automated methods processor for liquid chromatography. The methods processor enables the user to conduct a complete LC analysis from the injection of the sample to the printing of the answer completely automatically. The processor array programs and controls the analysis of up to 42 consecutive samples via a Perkin-Elmer cassette tape program. Additionally, the analyst can generate other programs from the SIGMA 10 keyboard. The analytical control features include pump method, data processor method, sample selection, sample sequence, and wavelength change. The software includes error messages, re-prompt for non-valid entries, and allowance for method modifications without a complete re-setup.



N-1337

AUTOMATED CAPILLARY GC

Convenience and ease of operation are incorporated in the Tracor 560 capillary gas chromatograph. Upon injection, a single automated control zeroes the baseline, initiates the oven temperature program, and times the inject period. This programmed control of the gas flow in the split mode results in a saving of carrier gas. When used in the splitless mode, it provides a precise purge delay time, essential for reproducibility. The injector allows both split and splitless injection techniques to be utilized without changing the inlets.

For further information concerning any of the news items, apply to the publisher, using the reply cards provided, quoting the reference number printed at the beginning of the item.

N-1330

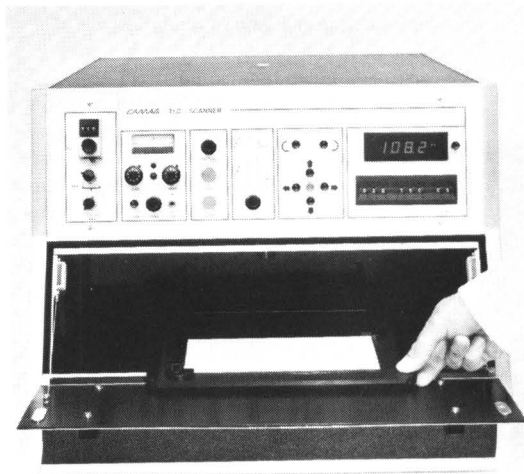
HIGH-TEMPERATURE, LOW-BLEED SEPTA FOR GC

A line of high-temperature, low-bleed septa for use in gas chromatography has been introduced by Hamilton. The new septa are called Hamilton Hi-Temp Low-Bleed Septa and are available with diameters ranging from 0.25 to 0.50 inch. Extensive testing has indicated low bleed characteristics of 2×10^{-12} A after multiple puncturing at 300° without irregular baseline shifts. The septa are available 12 to the package in a choice of nine diameters. They are made of siliconized rubber with Teflon backing, and require 30 minutes of conditioning for stabilization after installation.

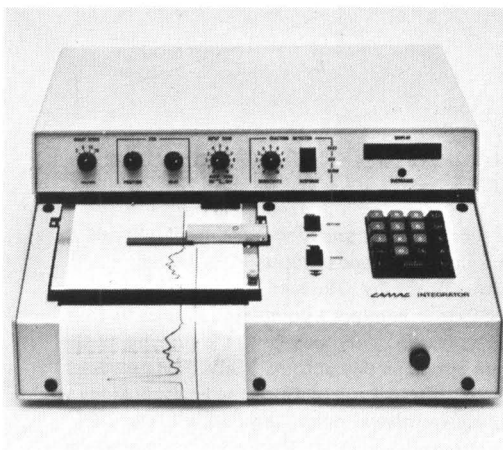
N-1329

NEW INSTRUMENTS FROM CAMAG

CAMAG's TLC/HPTLC scanner is now available with a monochromator. The effective range of the scanner is from 200 to 800 nm in absorption, fluorescence and fluorescence quenching modes. The whole chromatogram or segments of it are scanned automatically once the parameters have been selected.



The new CAMAG electrophoresis integrator is a microprocessor-controlled instrument coupled to a densitometer. It plots the analog curve and automatically prints out relative percentages, gram percentages, or pure integral values. Fraction limits are determined and marked. An editing feature allows the operator to selectively add or delete fraction limits and suppress entire fractions of the scan.



N-1328

GC SEPTA

Bulletin 780 from Supelco covers many interesting subjects concerning GC septa. In the Bulletin the manufacturer introduces the new septa of the Thermogreen LB-1 series. The Thermogreen LB-1 is a low-bleed, high-temperature septum. The new septum has less bleed at high temperatures than both the Blue HT and the old green septum from Supelco. The Thermogreen LB-1 has a good ability to resist leaks and the manufacturer ensures a high degree of batch-to-batch uniformity. The septum is designed to operate at inlet temperature settings of 200–300°. The septum is preconditioned as part of the manufacturing operation, and consequently, Supelco does not recommend solvent extraction or vacuum heating prior to use.

N-1332

COMPACT LIQUID CHROMATOGRAPH

The Schoeffel Instrument Division of Kratos, Inc. announces the availability of a compact high-performance liquid chromatograph, built around the company's LC 250/1 dual-piston pump. The complete chromatograph occupies less than 15 inches of bench space. The LC 250/1 is a constant-volume pump operating up to 6000 p.s.i. The pump provides accurate flow-rates between 0.1 and 9.9 ml/min. A solvent rack with column and injector brackets is featured in a space-saving design. A compartment below the

rack is provided for placement of the detector within only a few inches of the column outlet. The new chromatograph includes the LC 250/1 pump, the Model 7125 syringe loading sample injector, the SF 740 UV detector and the solvent/column rack.

N-1322

SHORT FORM CATALOG

In a 6 page brochure the Schoeffel Instruments Division of Kratos, Inc. briefly describes its complete line of HPLC components. These components may be used to upgrade existing liquid chromatographs or may be combined together to create a complete system. The new products in the catalog are: the Series 250 modular gradient chromatograph, two new absorbance detectors SF 740 and SF 771, and the FS 950 filter fluorometer.

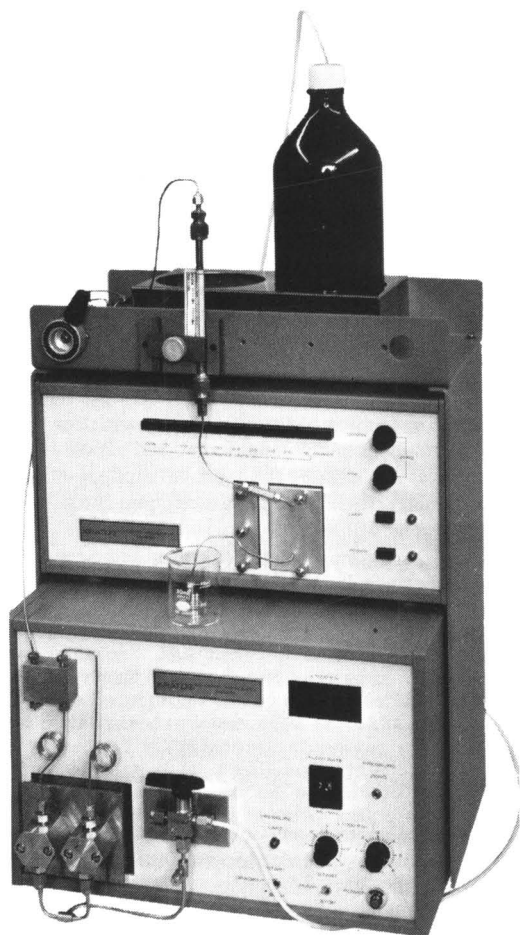
N-1320

NEW DETECTORS FROM DU PONT

The Model 860 UV/visible absorbance HPLC detector from Du Pont uses optical filters and a stable, low-pressure mercury vapour lamp source with a phosphor-coated tip. A two-position mounting permits easy conversion from 254 to 284 nm. The flow-cell design permits sensitivities to 0.002 absorbance units full scale. The sample absorbance is continuously displayed on a digital meter.

The Du Pont UV spectrophotometer detector is a compact variable-wavelength device, which uses a deuterium source and a dual-beam grating monochromator. Absorbance ranges are adjustable from 0.005 to 2.56 absorbance units full scale.

The third new detector is a refractive index detector. The unique flow cell of this detector is housed in a dewar flask. This eliminates the need for a liquid bath and minimizes drift caused by temperature changes. Two adjustable transparent plates are used to optically balance the light beam. Manual adjustment of the plates balances the light intensity of the two matched cadmium sulphide photocells.



N-1323

SUPELCO CHROMATOGRAPHY CATALOG 14

GPC COLUMNS

A versatile family of gel permeation chromatography columns, AquaporeTM, has been introduced by Chromatix for use by analytical and biochemists in the characterization of synthetic and biopolymers. Aquapore columns can be used to separate polymers soluble either in aqueous media as well as polar or non-polar solvents. Aquapore columns are available in a range of pore sizes suitable for separating materials over a molecular weight range of several hundred to about five million.

Supelco has released its 1979 international catalog 14. It provides a 24-page applications section with information on many areas of gas chromatography. In addition it offers columns, column packings, accessories, derivatizing reagents, TLC accessories, lipid chemicals, textbooks, etc. Among the new products are improved column packings for clinical and environmental analysis, micro- and macro-autoclavable dispensers, septums, HPLC valves and syringes, capillary adapter kits and control modules. The catalog also lists 47 technical bulletins available from the manufacturer.

MEETINGS

CHROMATOGRAPHY '80

15TH INTERNATIONAL SYMPOSIUM ON ADVANCES IN CHROMATOGRAPHY

The 15th International Symposium on Advances in Chromatography will be held October 6-9, 1980 in Houston, Texas, U.S.A. The scope of the meeting will cover invited papers, poster sessions and informal discussion groups by outstanding researchers from throughout the world in all fields of chromatography. In particular, new developments in gas, liquid and high-performance thin-layer chromatography will be included. There will also be a commercial exhibition of the latest instrumentation and books. This symposium will be held in conjunction with Expochem '80, an international exposition of analytical instruments for the industrial and biomedical fields. Short courses in (a) capillary gas chromatography, (b) gas chromatography-mass spectrometry, (c) high-performance liquid chromatography and (d) high-performance thin-layer chromatography will be held on the weekend prior to the symposium. Authors desiring to present poster session papers must submit 200-word abstracts by April 1, 1980. Program and registration forms will be available in June. All correspondence concerning the symposium and short courses should be directed to Professor A. Zlatkis, Chemistry Department, University of Houston, Houston, Texas 77004, U.S.A.

13TH INTERNATIONAL SYMPOSIUM ON CHROMATOGRAPHY

In addition to the news announcement published in Vol. 172, the Organizing Committee of the 13th International Symposium on Chromatography, to be held in Cannes from June 30-July 4, 1980, informed us of the following:

The first week of July is the beginning of the high season in Cannes. It will be nearly impossible to place a hotel reservation for this period by early Spring. Furthermore, there are only a limited number of rooms (about 650) available at Congress prices, until late January. Accordingly, it is strongly advised to register as soon as possible and at any rate before March 1, 1980. Registration and hotel reservation forms are available from GAMS, 88 Boulevard Malesherbes, 75008 Paris, France. Tel. (1) 563.93.04.

GENERAL INFORMATION

(A leaflet *Instructions to Authors* can be obtained by application to the publisher.)

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- 2 L. R. Snyder, *Principles of Adsorption Chromatography*, Marcel Dekker, New York, 1968, p. 201.
- 3 H. C. S. Wood and R. Wigglesworth, in S. Coffey (Editor), *Rodd's Chemistry of Carbon Compounds*, Vol. IV, *Heterocyclic Compounds*, Part B, Elsevier, Amsterdam, Oxford, New York, 2nd ed., 1977, Ch. 11, p. 201.
- 4 E. C. Horning, J.-P. Thenot and M. G. Horning, in A. P. De Leenheer and R. R. Roncucci (Editors), *Proc. 1st Int. Symp. Quantitative Mass Spectrometry in Life Sciences*, Ghent, June 16–18, 1976, Elsevier, Amsterdam, Oxford, New York, 1977, p. 1.

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