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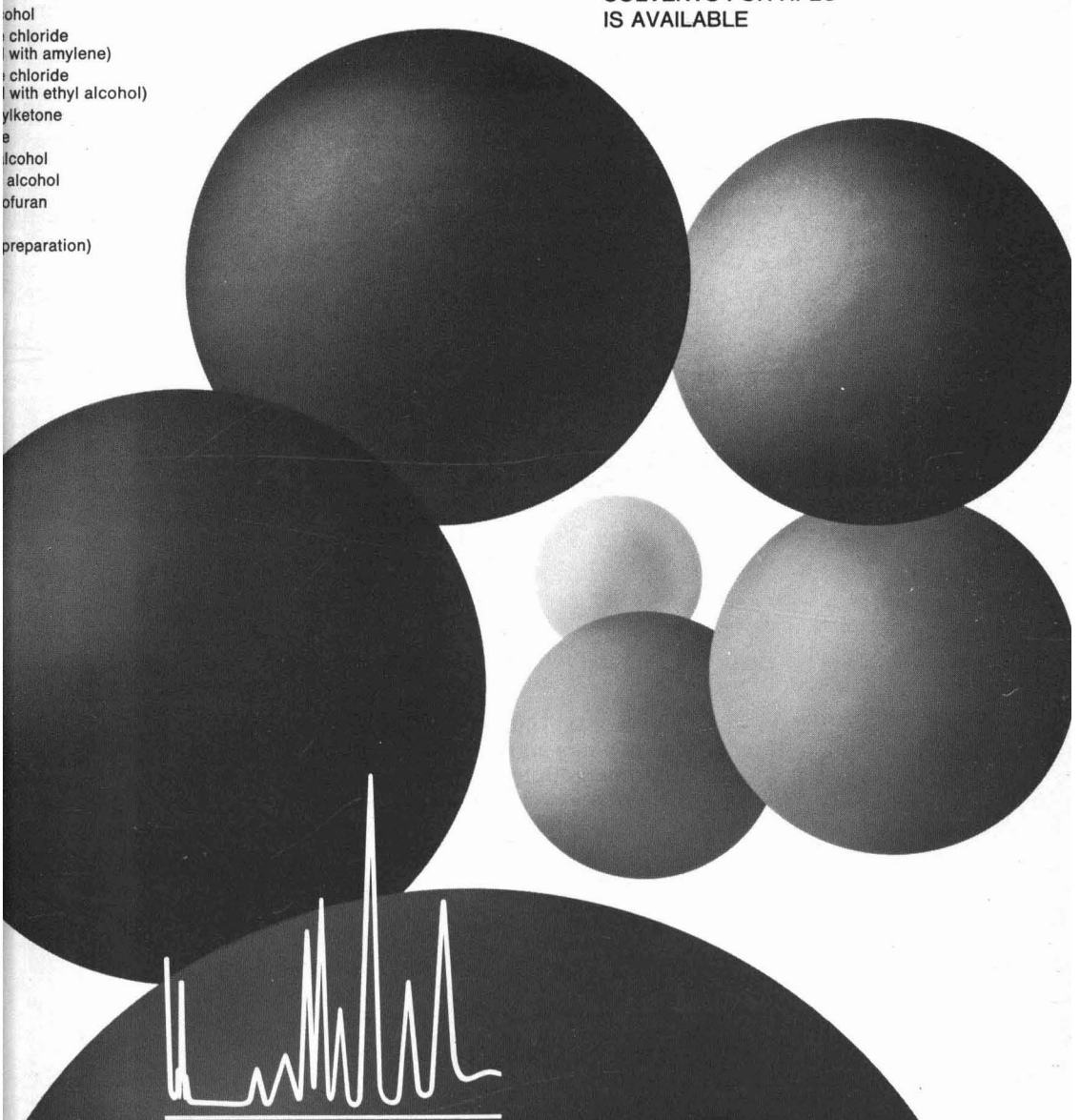

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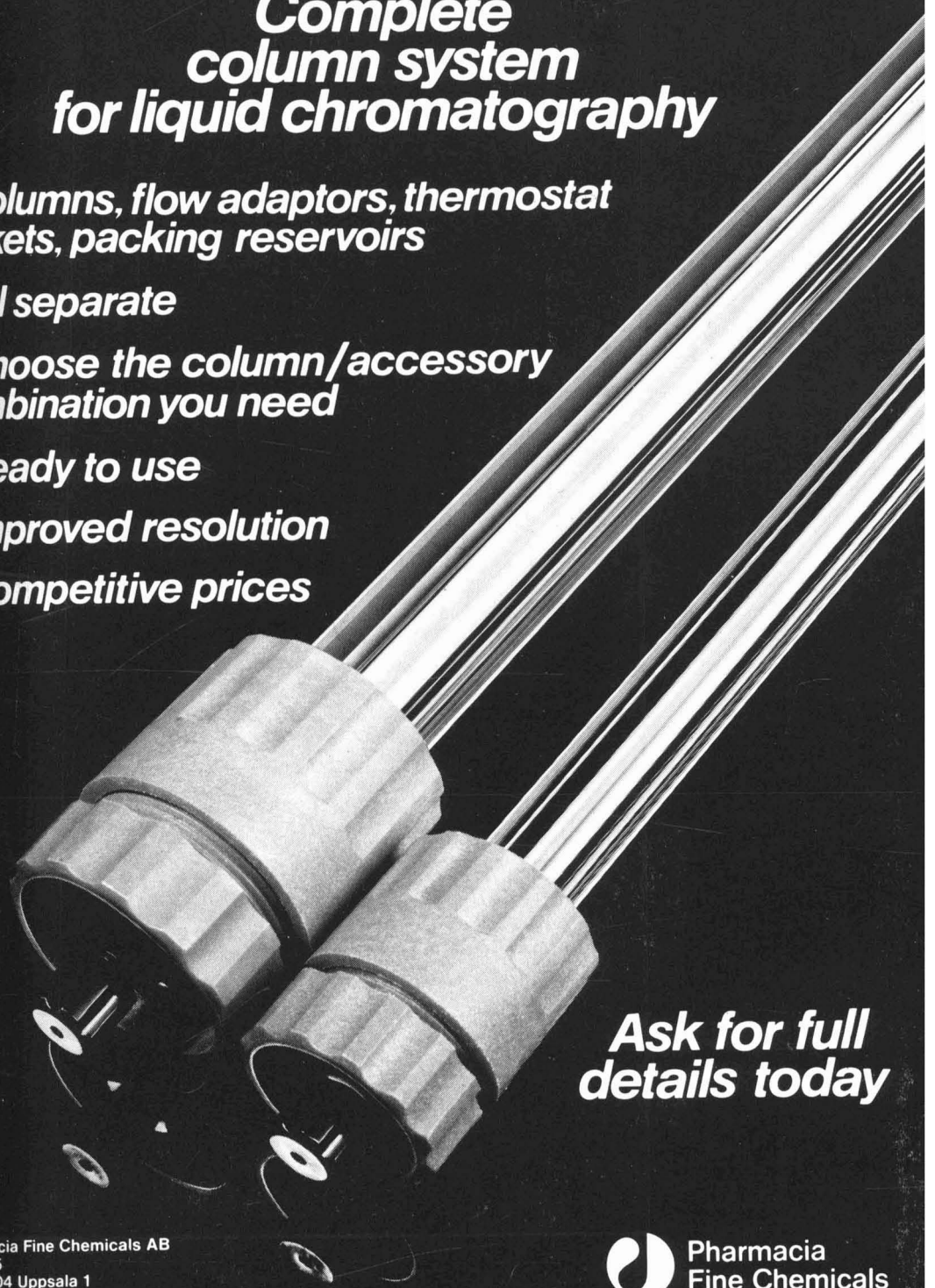

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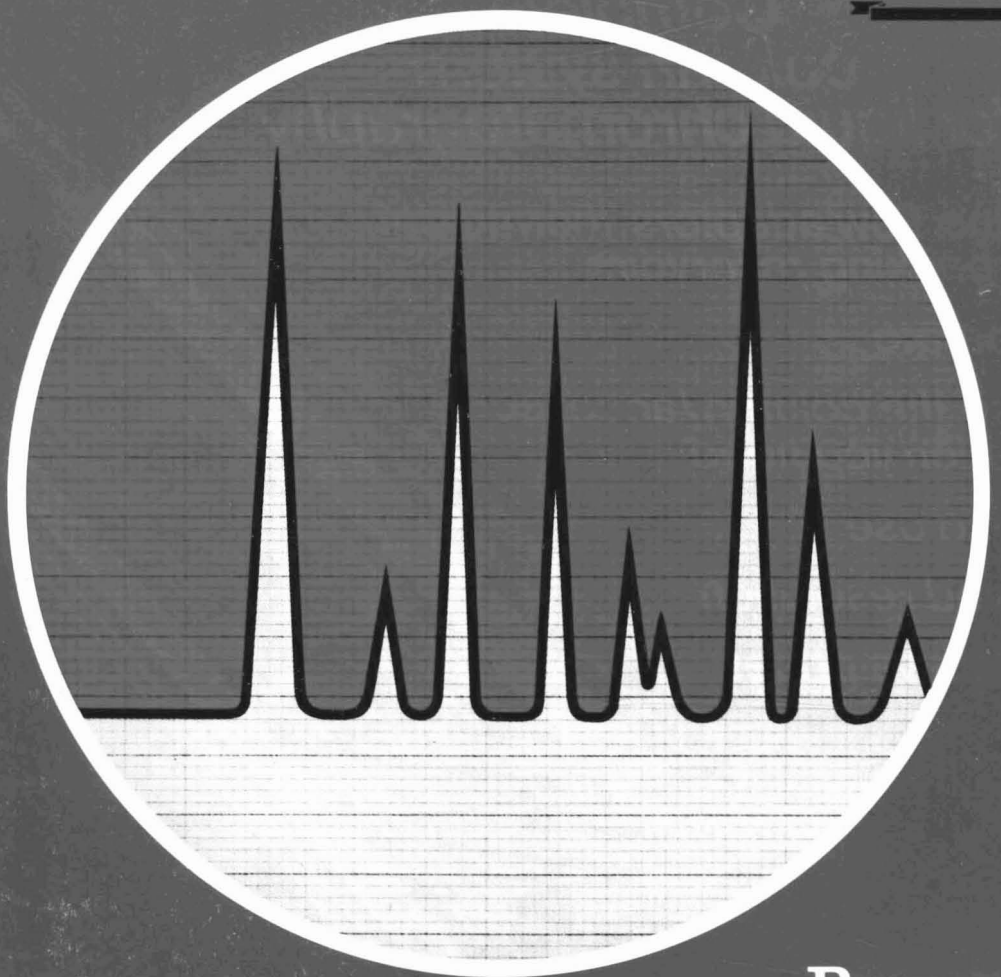

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# NEW CONTINUOUS EXTRACTION METHOD WITH A COIL PLANET CENTRIFUGE

YOICHIRO ITO

*Laboratory of Technical Development, National Heart, Lung and Blood Institute, Bethesda, MD 20205 (U.S.A.)*

(Received July 16th, 1980)*

## SUMMARY

A compact table-top model of the coil planet centrifuge simultaneously enables both preliminary purification and enrichment of samples from crude extracts or biological fluids. The method uses hydrodynamic behavior of two immiscible solvent phases in a rotating coiled tube to retain the stationary phase against a high flow-rate of mobile phase. Consequently, a small quantity of the sample present in a large volume of mobile phase is efficiently extracted into a small volume of the stationary phase within a short period of time and at a high recovery rate. The capability of the present method was demonstrated in the extraction of dinitrophenylamino acids (used as a comparative performance standard) with a set of two-phase solvent systems composed of ethyl acetate and 0.5 *M* $NaH_2PO_4$.

## INTRODUCTION

Preliminary cleaning-up of crude extracts or biological fluids is often essential for purification of biological materials. When a small quantity of the material of interest is present in a relatively large quantity of the solvent, enrichment is also necessary. Conventional procedures such as repetitive extraction with a separatory funnel or a Craig countercurrent distribution method usually result in a large quantity of harvested solvent which necessitates further concentration.

The present paper introduces an efficient extraction method utilizing a coil planet centrifugation. The method enables both cleaning-up and enrichment in a short period of time and at a high recovery rate. The capability of the method was demonstrated on extraction of small amounts of dinitrophenyl (DNP) amino acids from several hundred milliliters of either aqueous or non-aqueous phase composed of ethyl acetate and 0.5 *M* sodium phosphate aqueous solution.

## PRINCIPLE

The method takes advantage of the intriguing hydrodynamic behavior of two immiscible solvent phases in a coiled tube rotating in an acceleration field. When a

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coiled tube is slowly rotated around its horizontally oriented axis, particles present in the coil move toward one end of the coil. This end is defined as the head and the other end as the tail of the coil. Two immiscible solvents confined in such a tube are usually distributed from the head toward the tail at a particular volume ratio and any excess of either phase remains at the tail of the coil. While the distribution ratio of the two solvents varies with a number of parameters, the rotational speed of the coil becomes the major determinant for the phase distribution of a given pair of solvents. At a very slow rotational speed the two phases are distributed so that they are nearly in equal amount in each coiled turn. At a very high revolutional speed a strong centrifugal force field separates the two phases in such a way that the heavier phase occupies the outer portion and the lighter phase the inner portion of each helical turn. This results in the distribution ratio of the two phases in each helical turn being equal to the volume ratio originally present in the coil. However, when the rotational speed is between these two extremities, one of the phases occupies more space in the coil on the headside and in some cases the two phases are completely separated in the coil, *i.e.*, one phase entirely occupies the head side and the other phase the tail side of the coil. Ideal two-phase distribution for continuous extraction is represented by this complete phase separation at this intermediate rotational speed.

Let us assume a pair of immiscible solvent phases A and B where phase A is distributed on the head side and phase B on the tail side of the coil. Under this particular circumstance, continuous extraction is possible in three ways. In the first method, the coil is filled with phase A (stationary phase) followed by elution with phase B (mobile phase containing the sample) through the head end of the coil. Phase B then travels through phase A in the coil toward the tail. Consequently, the sample present in phase B is extracted into the stationary phase A and the stripped phase B is eluted through the tail of the coil. In the second operation, the coil is first filled with phase B (stationary phase) and phase A (mobile phase containing the sample) is pumped through the tail of the coil. Extraction process similarly takes place in the coil, the stripped phase A being eluted through the head of the coil. The third operation involves dual countercurrent extraction (not described in this paper) in which phases A and B are simultaneously introduced into the coil through the tail and the head, respectively. In this case the coil should be equipped with an additional pair of flow tubes at each end to collect both enriched and stripped phases. If desirable, the sample solution may be fed into the coil through another flow line connected at the middle portion of the coil.

As mentioned earlier, these extraction methods are perfected by providing an operational condition where the two phases are separated completely along the length of the coil. Use of the rotating coil in the gravitational field, however, usually fails to produce this ideal type of hydrodynamic behavior of the two phases. The search for a suitable extraction scheme which yields complete phase separation in a coiled tube has been successful in the utilization of a coil planet centrifuge. The apparatus provides a particular mode of the synchronous planetary motion to a coiled tube, *i.e.*, the coil revolves around the central axis of the centrifuge and rotates about its own axis at the same angular velocity and in the same direction[1–3].

The centrifugal force field produced by this planetary motion[3,4] is highly dependent upon the location of the point on the holder which is conveniently expressed by $\beta$, *i.e.*, the ratio between the radii of rotation and revolution. When the $\beta$ value

exceeds 0.25, the centrifugal force vector is always directed outwardly from the holder while it oscillates in both amplitude and direction during each revolutional cycle of the holder. Though the motion of the two-phase solvent in the coil subjected to such a centrifugal force field is hardly predictable, the behavior of the two phases can be easily observed through the tube wall under stroboscopic illumination.

A series of observations has been made on various two-phase solvent systems having a wide spectrum of physical properties. The results so far obtained indicate that the distribution of the two phases is affected by three major factors, *i.e.*, wall affinity, relative density and viscosity of the two phases. The phase which has higher wall affinity, lower density and less viscosity tends to distribute itself toward the head of the coil. When all three requirements are satisfied, the upper phase will quickly move toward the head and the phase separation is completed in a short period of time. This ideal group of the solvent pair includes (if PTFE tube is used as the column) a number of useful extraction media such as hexane, ether, ethyl acetate, toluene, methyl ethyl ketone, benzene, etc., mixed with aqueous solution where various salts can be added to adjust the pH and ionic strength of the aqueous phase. Various third solvents such as methanol, acetic acid, etc., can also be added without altering the overall behavior of the two phases.

When the two solvent phases fail to meet the above requirements, complete phase separation may not occur; instead one of the phases usually dominates at the head side of the coil. Two typical solvent pairs in this group have been tested. In a *n*-butanol–water system the non-aqueous phase is more viscous than the lower aqueous phase. In this case the distribution ratio of the two phases is greatly affected by the $\beta$ values. At $\beta = 0.25$, the aqueous phase is almost entirely distributed on the head side while at $\beta = 0.75$ the non-aqueous phase dominantly occupies the head of the coil especially under a high revolutional speed. In a chloroform–water system, the non-aqueous phase is much heavier than the aqueous phase. Probably due to this great difference in density, the aqueous phase always dominates on the head side regardless of the $\beta$ values. In these non-ideal solvent pairs, application is limited to head–tail elution using the dominant phase as the stationary phase.

## EXPERIMENTAL

### *Apparatus*

The design of the apparatus used in the present studies is similar to the toroidal coil planet centrifuge which permits continuous elution without the use of rotating seals as described earlier[2,3]. Fig. 1 shows the cross-sectional view of the apparatus. The motor (Electro-Craft) drives the rotary frame around the horizontal stationary pipe (shaded) mounted on the axis of the centrifuge. The rotary frame consists of a pair of aluminum discs rigidly bridged together with multiple links (not shown in the figure) and holds a pair of rotary column holders in the symmetrical positions 10 cm away from the central axis of the centrifuge. The bottom holder has a diameter of 15 cm ($\beta = 0.75$) and the top holder of 10 cm ($\beta = 0.5$). The shaft of each holder is equipped with a plastic planetary gear which is coupled to an identical sun gear (shaded) mounted around the central stationary pipe. In order to provide mechanical stability, a short coupling pipe is coaxially mounted to the free end (right side) of the rotary frame while the other end of the coupling pipe is supported by a stationary wall member of the centrifuge through a ball bearing. The coiled column

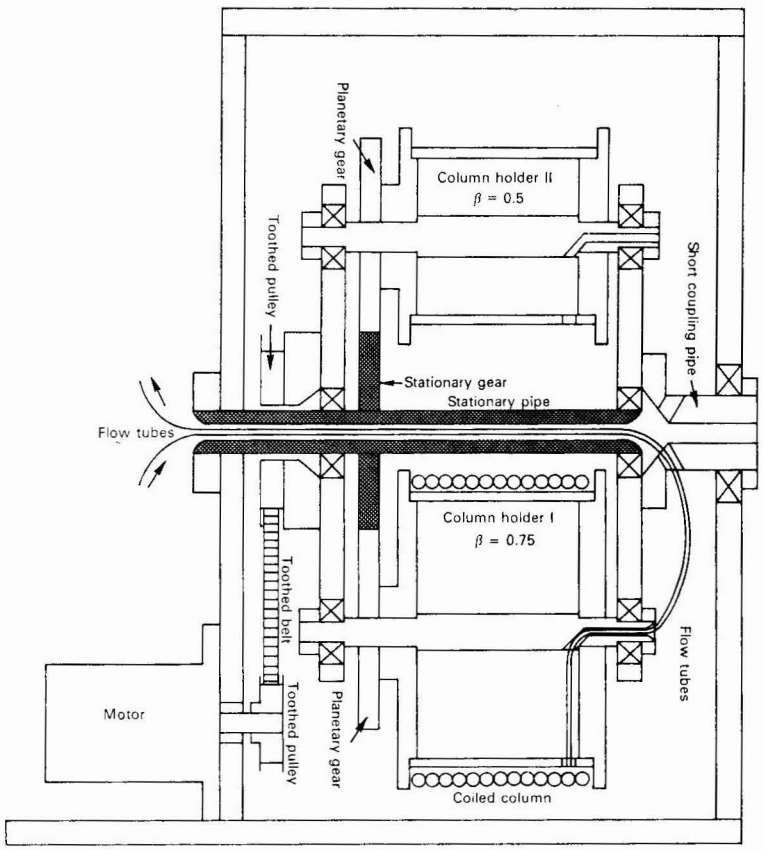


Fig. 1. Cross-sectional view of the apparatus.

was made by winding the desired length of a PTFE tube around one of the holders while a counterweight is applied on the other holder to balance the centrifuge. A pair of flow tubes from the coiled column is first passed through the center hole of the holder shaft and led through the side hole of the short coupling pipe to reach the opening of the central stationary pipe. These flow tubes are thoroughly lubricated with silicone grease and protected with a piece of plastic tubing at each supported portion to prevent direct contact with metal parts. The revolutional speed can be regulated up to 1000 rpm. The apparatus is a compact table-top model whose dimensions are *ca.* 16 × 16 × 17 in.

*Studies on retention of the stationary phase*

The capability of the present scheme in retaining a large amount of the stationary phase was demonstrated with a coiled column prepared from 2.5 m × 2.6 mm I.D. PTFE tube (Zeus Industrial Products, Raritan, NJ, U.S.A.) which was coiled around a holder having a $\beta$ value of 0.75. The column consisted of five helical turns and had a total capacity of *ca.* 15 ml. Typical two-phase solvent systems composed of ethyl acetate–water and ethyl acetate–0.5 *M* sodium phosphate (pH 4.4) at a volume ratio of 1:1 were selected. Each two-phase system was equilibrated in a

separatory funnel and separated before use. In each operation the coiled column and the free space in the flow path were entirely filled with the stationary phase and the mobile phase was pumped into the column in the proper direction (head–tail elution for the aqueous phase and tail–head elution for the non-aqueous phase) while the apparatus was run at a given revolutional speed. The eluate through the outlet of the column was pooled in a graduated cylinder to measure the volume of the eluted stationary phase, $V_s$. From the predetermined figures of the total column capacity, $V_c$, and the free space in the flow path, $V_f$, the percentage retention, $R$, of the stationary phase relative to the total column capacity was calculated according to the formula: $R = 100\,(V_c + V_f - V_s)/V_c$. The experiments were performed under various revolutional speeds and flow-rates using both non-aqueous and aqueous phases as the stationary phase.

*Continuous extraction experiments*

A series of experiments has been performed to demonstrate the capability of the present scheme to extract a solute present in a large volume of the mobile phase into a small volume of the stationary phase retained in the coiled column. This requires a set of conditions such that the solute must favor partition to the stationary phase. With commonly used extraction media such as an ethyl acetate–aqueous system, partition coefficients of various biological materials can be conveniently adjusted by modifying the pH and/or ionic strength of the aqueous phase to meet the above requirement. For the present studies, a pair of DNP-amino acids (Sigma, St. Louis, MO, U.S.A.), N-DNP-L-leucine (DNP-Leu) and delta-N-DNP-L-ornithine (DNP-Orn), were selected as samples because they are readily observed through the column wall during the extraction process under stroboscopic illumination and also provide suitable partition coefficients for this present solvent system. The experiments were performed with the coiled column used in the previous retention studies. The overall experimental conditions in the following studies are summarized in Table I.

A typical extraction procedure may be divided into three steps, *i.e.*, extraction, cleaning, and collection. In each operation, the column was filled with the stationary phase and the mobile phase containing the sample was eluted through the column in the proper direction while the apparatus was run at 600 rpm. The extraction process was continued until 400 ml of the mobile phase was eluted. Then the mobile phase was replaced by the same phase but free of solute to wash the column contents. This cleaning process was continued until the additional 100 ml of the mobile phase was eluted. This would elute out all impurities having partition coefficients of 0.1 or greater. The sample extracted into the stationary phase in the coiled column was collected by eluting with the mobile phase in the opposite direction. This was done by switching the feed and return flow lines either by simply disconnecting the flow lines or the use of a four-way slide valve (Pierce, Rockford, IL, U.S.A.). The sample still remaining in the column was then washed out by eluting the column with the other phase originally used as the stationary phase.

Sample collection from the column may be performed in different ways. When the mobile phase is the aqueous phase, modification of the pH and/or ionic strength often results in a great shift in the partition coefficient of the solute in such a way that the solute favors partition into the aqueous phase. In this case either stepwise or gradient elution with such a modified aqueous phase produces a chromato-

TABLE I

SUMMARY OF EXPERIMENTAL CONDITIONS AND RESULTS FOR CONTINUOUS EXTRACTION

| *Exp. No.* | *Solvent system* | *Mobile phase* | *Stationary phase* | *Sample (P.C.)** | *Sample concn. in mobile phase (mg%)* | *Extracted mobile phase volume (ml)* | *Flow-rate (ml/h) (direction)* | *rpm* | *Collected stationary phase volume (ml)* | *Sample recovery (%)* |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Ethyl acetate–0.5 *M* $NaH_2PO_4$(1:2) | Aqueous | Non-aqueous | DNP-Leu (<0.01) | 4 | 400 | 516 (head–tail) | 600 | 10.5 | 94 |
| 2 | Ethyl acetate–0.5 *M* $NaH_2PO_4$(1:2) | Aqueous | Non-aqueous | DNP-Leu (<0.01) | 0.4 | 400 | 516 (head–tail) | 600 | 10.0 | 97 |
| 3 | Ethyl acetate-0.5 *M* $NaH_2PO_4$(1:2) | Aqueous | Non-aqueous | DNP-Leu (<0.01) | 0.04 | 400 | 516 (head–tail) | 600 | 10.4 | 100 |
| 4 | Ethyl acetate–0.5 *M* $NaH_2PO_4$(2:1) | Non-aqueous | Aqueous | DNP-Orn (<0.01) | 0.4 | 400 | 516 (tail–head) | 600 | 11.8 | 97 |
| 5 | Ethyl acetate–0.5 *M* $NaH_2PO_4$(2:1) | Non-aqueous | Aqueous | DNP-Orn (<0.01) | 0.04 | 400 | 516 (tail–head) | 600 | 11.8 | 100 |
| 6 | Non-equilibrium system | 5% Ethyl acetate in 0.5 *M* $NaH_2PO_4$ | Ethyl acetate | DNP-Leu (<0.01) | 0.4 | 400 | 516 (head–tail) | 600 | 6.1 | 99 |

* Partition coefficient (P.C.) is defined as solute concentration in the mobile phase divided by that in the stationary phase.

graphic separation of the solute which can be monitored by a conventional UV detector. This results in further purification of the solute retained in the column.

The degree of sample recovery was estimated by comparing the amount of the sample in the original mobile phase to that in the collected stationary phase. A Beckman DU spectrophotometer was used to measure the absorbance at 430 nm.

## RESULTS AND DISCUSSION

The results of the retention studies are summarized in Fig. 2, where the percentage retention of the stationary phase was plotted against the applied revolutional speeds. The three lines drawn in each diagram indicate the effects of the different flow-rates applied. The flow-rate of 516 ml/h is the maximum rate available with the Beckman Accu Pump employed. The ideal retention level for extraction may be considered to be over 70% at or near the plateau of the curve, although much lower retention levels can be applied for extraction unless carryover of the stationary phase occurs. Fig. 2A and B show the retention curves of the solvent system com-

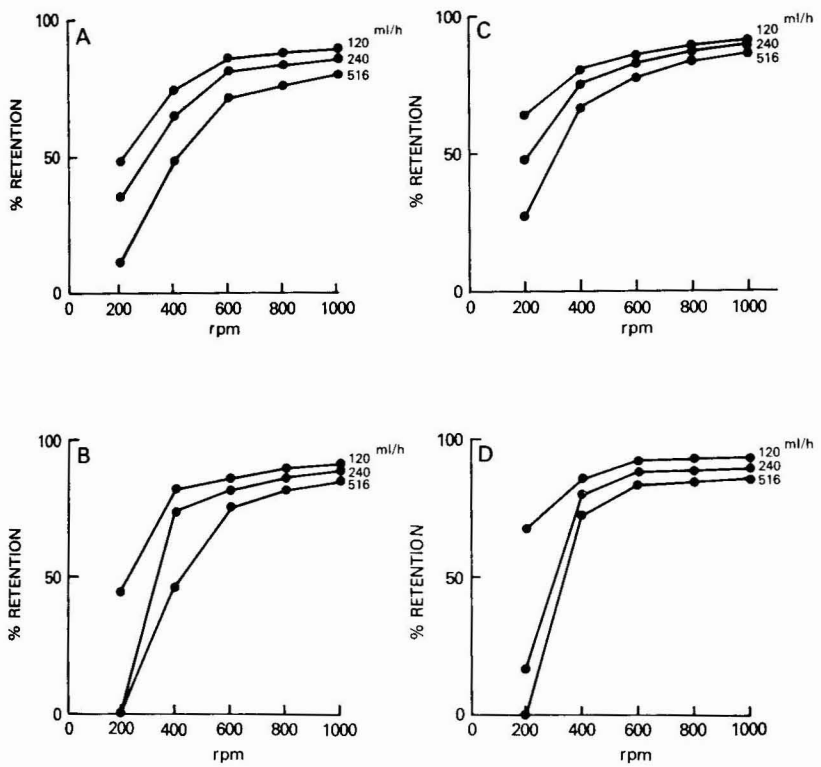


Fig. 2. Effects of revolutional speed and flow-rate on retention of the stationary phase. A, Phase system: ethyl acetate–water; stationary phase: upper non-aqueous phase; elution: head–tail. B, Phase system: ethyl acetate–water; stationary phase: lower aqueous phase; elution: tail–head. C, Phase system: ethyl acetate–0.5 *M* $NaH_2PO_4$ (1:1); stationary phase: upper non-aqueous phase; elution: head–tail. D, Phase system: ethyl acetate–0.5 *M* $NaH_2PO_4$ (1:1); stationary phase: lower aqueous phase; elution: tail–head.

posed of ethyl acetate and water, where both non-aqueous (A) and aqueous (B) phases were used as the stationary phase. In both cases the ideal retention levels are provided at the revolutional speed of over 600 rpm at all flow-rates applied. Comparison between A and B reveals that the retention levels of the aqueous phase in the tail–head elution always exceed those of the non-aqueous phase in the head–tail elution. This may be due to the higher wall affinity and lower viscosity of the non-aqueous phase which provides less resistance against the flow.

Fig. 2C and D show similar retention curves for the phase system composed of ethyl acetate–0.5 *M* $NaH_2PO_4$ (1:1). In both C and D, retention levels show much improvement over the previous phase system. Addition of salt to the phase system results in a greater density difference which promotes movement of the phases in the coil as described earlier. The overall results indicate that the system provides excellent retention under a broad range of operational conditions for both aqueous and non-aqueous stationary phases. The results also suggest that much higher flow-rates are applicable with high revolutional speeds.

In order to demonstrate the extraction capability of the present scheme, a series of model experiments has been performed with sets of solvent systems and DNP-amino acid samples as shown in Table I. In experiments 1–3 in Table I, DNP-Leu was dissolved in 400 ml of the aqueous phase at various concentrations and extracted into the non-aqueous phase retained in the column. The extracted sample was then cleaned by eluting the column with 100 ml of the clean aqueous phase and then collected from the column. The harvested stationary phase volume measured *ca.* 10 ml, containing over 90% of the original sample. A small amount of the sample still remaining in the column, usually a few percents of the total, was conveniently recovered by eluting the column with several milliliters of the non-aqueous phase. The total sample recovery is always well over 90%, as shown in the table. The reduction of the sample concentration from 4 mg% to 0.04 mg% somewhat improved the recovery rate, indicating that no significant sample loss occurs due to the adsorption effects and that further reduction of the sample concentration is feasible with high levels of recovery. The mode of elution that uses the non-aqueous phase as the stationary phase renders a great advantage in practical extraction in that the collected solvent is highly volatile and free of salts, facilitating further concentration. It also permits the stepwise or gradient elution of the sample by eluting the column with a modified aqueous phase to achieve further purification.

In experiments 4 and 5 in Table I, the DNP-Orn sample was dissolved in 400 ml of the mobile non-aqueous phase and extracted into the stationary aqueous phase by eluting the column from the tail toward the head. The retained aqueous phase in the column was then similarly cleaned with 100 ml of non-aqueous phase free of sample. The collected stationary aqueous phase measured *ca.* 12 ml in volume. This exceeds the volumes in experiments 1–3, as expected from the results of the retention studies. The sample still remaining in the column was eluted out with several milliliters of the aqueous phase. The sample recovery ranged over 95% with an improved figure at the reduced sample concentration as observed in the previous experiments.

In practice, application of the method to aqueous crude extracts or physiological fluids requires a preliminary adjustment of the solvent composition to provide a suitable partition coefficient of the desired material for the applied pair of solvents.

In this case pre-equilibration of the two phases is not essential. Experiment 6 in Table I shows an example of operation with such non-equilibrated solvents. The sample DNP-Leu was first dissolved in 400 ml of 0.5 *M* $NaH_2PO_4$ aqueous solution containing ethyl acetate at 5%, which is slightly below the saturation level of *ca.* 7%. The column was filled with ethyl acetate followed by elution with the above sample solution. Both extraction and cleaning processes were performed as in other experiments. The sample solution collected from the column measured slightly over 6 ml. This depletion of the stationary phase apparently resulted from use of the non-equilibrated solvent pair but without any effect on the sample recovery.

The overall results indicate a potential usefulness of the present method in processing a large amount of crude extracts or biological fluids in research laboratories. A small amount of the sample present in several hundred milliliters of the original solution can be enriched in 10 ml of the non-aqueous phase free of salt in 1 h at a high recovery rate.

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# PREPARATIVE COUNTER-CURRENT CHROMATOGRAPHY WITH A ROTATING COIL ASSEMBLY

YOICHIRO ITO* and ROHIT BHATNAGAR

*Laboratory of Technical Development, National Heart, Lung, and Blood Institute, Bethesda, MD 20205 (U.S.A.)*

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## SUMMARY

We have designed a simple bench top model of a counter-current chromatograph which performs efficient preparative separations without the use of solid supports. The stationary phase is retained by gravity in a large diameter coil which rotates to promote efficient mixing of the two phases. Continuous elution of the mobile phase is accomplished without the use of rotating seals. We demonstrated the efficiency of the system by separating gram quantities of dinitrophenyl amino acids. The design and construction of the apparatus should permit easy increases in scale for industrial applications.

## INTRODUCTION

In the past, several devices for performing preparative-scale counter-current chromatography have been developed[1–10]. Among those schemes the most efficient separations have been obtained from schemes which employ a rotating coiled column in an acceleration field[2–10]. Efforts have been successfully made to eliminate the use of rotating seals in utilizing the horizontal flow-through coil planet centrifuges[4–10]. However, these devices hold a preparative column assembly on one side of the rotary arm and, therefore, application of a large preparative column requires a fair amount of laboratory space.

This paper describes a new preparative counter-current chromatographic scheme which compactly holds a large coil assembly at the center of the apparatus and is amenable for further scaling-up of the sample loading capacity for industrial separations. The partition capability of the present scheme was demonstrated by the separation of a set of dinitrophenyl (DNP) amino acids with a two-phase solvent system composed of chloroform–acetic acid–0.1 *N* hydrochloric acid at a volume ratio of 2:2:1.

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## PRINCIPLE AND DESIGN OF THE APPARATUS

The present scheme uses a coiled tube which slowly rotates around its horizontal axis with respect to the gravitational field. Particles introduced in such a coil move toward one end of the coil. This end is defined as the head and the other end, the tail of the coil. A two-phase solvent system confined in this rotating coil distributes itself in such a way that nearly equal volumes of the two phases occupy each helical turn while any excess of either phase remains at the tail. This hydrodynamic behaviour of the solvents allows elution of either phase through the head while retaining a large amount of the stationary phase in each turn of the coil. Consequently, solutes introduced through the head of the coil are subjected to an efficient partition process between the mobile and stationary phases in each turn of the coil and are eluted through the tail in the order of their partition coefficients as in liquid chromatography but in the absence of solid supports.

Applications of this scheme requires a flow-through mechanism to elute the solvent through the rotating coil. The simple rotary coil assembly introduced here is equipped with a rotary-seal-free flow-through system which eliminates various complications such as leakage, corrosion and contamination caused by the use of rotating seals.

Fig. 1 shows a simplified cross-sectional view through the central axis of the apparatus. The motor drives a rotary frame which consists of three aluminum arms rigidly bridged together with links. The frame holds two rotary elements, the countershaft and the centrally located column holder assembly. The countershaft is equipped with a toothed pulley at one end and a plastic gear at the other end. The pulley of the countershaft is coupled with a toothed belt to an identical stationary pulley mounted on the stationary wall member of the apparatus. This coupling causes a counter-rotation of the countershaft on the rotary frame. This motion is further

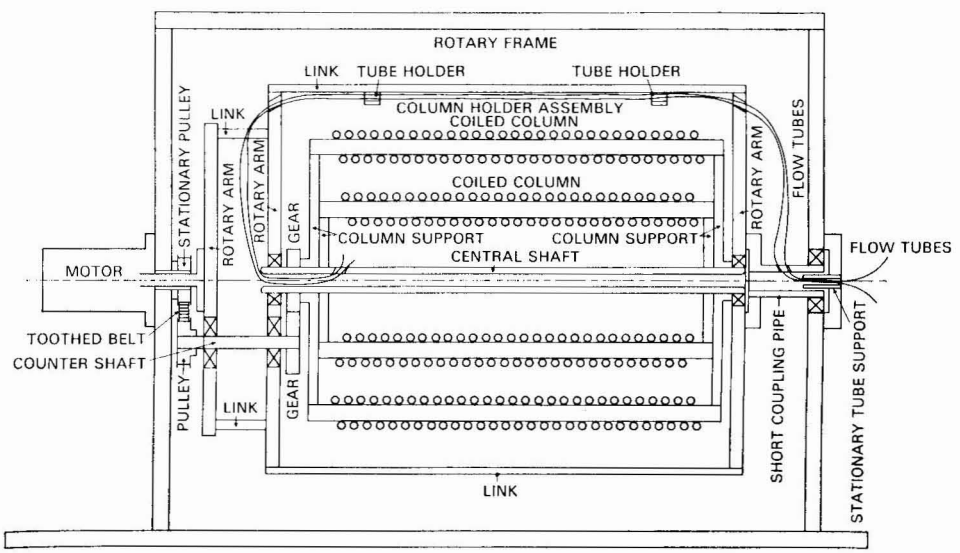


Fig. 1. A simplified cross-sectional view through the central axis of the apparatus.

conveyed to the central column holder assembly by 1:1 gear coupling. Consequently, the column holder assembly rotates around its own axis at a rate twice that of the rotary frame in the same direction. This particular design gives a great advantage in that the scheme allows the flow in and out of the rotating column without the use of rotating seals[11–13].

Separation columns used in the present studies consist of coiled glass tubes of 0.5 cm I.D., with different helical diameters (Kontes, Vineland, NJ, U.S.A.). One column has a 2.5-cm helical diameter with a 90-ml capacity and the other has a 1.25-cm helical diameter with a 45-ml capacity. Both columns contain approximately 50 helical turns. Each column is supported by a hollow aluminum core of the suitable diameter which is in turn mounted onto the column holder by a screw at each end. The column holder is equipped with two different levels for mounting columns, the first level being located 6.5 cm from the central axis of the apparatus and the second level, 13 cm from the same axis. A maximum of 30 columns can be mounted to the holder, 10 columns at the first level and 20 columns at the second level. The desired number of columns can be connected in series with a short piece of heat shrinkable PTFE tubing (Zeus Industrial Products, Raritan, NJ, U.S.A.) at each junction.

Flow tubes from the column are first led through the center hole of the column holder shaft, then passed through a pair of holes at the periphery of the rotary arms, and finally supported by a stationary tube support located at the central axis of the apparatus. These tubes are thoroughly lubricated with silicone grease and protected with a piece of plastic tubing to prevent contact with metal parts.

The rotational speed of the column assembly can be regulated up to 300 rpm. However, in the present studies, fragility of the glass column limits the maximum rate down to approximately 100 rpm. A Beckman Accu pump and Chromatronix pump are used to elute the solvents and an LKB Uvicord III to monitor the eluate at 280 nm.

## EXPERIMENTAL

### *Preliminary studies on partition capability*

The performance of the present counter-current chromatographic scheme was investigated by measuring the degree of stationary phase retention and partition efficiency. The two types of coils with 1.25 and 2.5 cm O.D. cores were tested, each mounted in both inner and outer positions of the column holder.

The degree of retention of the stationary phase in each column was measured with a two-phase solvent system composed of chloroform–acetic acid–water at a 2:2:1 volume ratio under various rotational speeds and flow-rates. The two-phase solvent system was first equilibrated in a separatory funnel at room temperature and separated before use. In each measurement the column was entirely filled with the mobile phase, either upper aqueous or lower non-aqueous phase. Then a given volume of the stationary phase which occupies "*A*" helical turns of the column was introduced through the head of the column. In order to visualize the stationary phase, a small amount of dye which favors partition to the stationary phase was dissolved in the stationary phase. Sudan III was used to color the non-aqueous phase and acid fuchsin to color the aqueous phase. Then the mobile phase was pumped through the head of the column while the column was rotated at a given rate. The two phases soon reached hydrodynamic equilibrium and the number of helical turns "*B*" containing the colored

stationary phase was read. The percentage retention relative to the total column capacity was obtained by the simple expression, $100A/B$. The measurement can be repeated by changing rotational speed or flow-rate without renewing the column contents until carryover of the stationary phase occurs.

The partition efficiency of each column was evaluated with a two-phase solvent system composed of chloroform–acetic acid–0.1 *N* hydrochloric acid at a 2:2:1 volume ratio and a pair of DNP amino acids as test samples. The two-phase solvent system was equilibrated in a separatory funnel at room temperature and separated before use. The sample solution was prepared by dissolving N-DNP–DL-glutamic acid (DNP-glu) and N-2,4-DNP–L-alanine (DNP-ala) (Sigma, St. Louis, MO, U.S.A.) in the upper aqueous phase to obtain the 0.5 g% concentration of each component. In each separation the column was first filled with the stationary phase. This was followed by injection of 0.5 ml of the sample solution through the sample port located on the flow line between the pump and the inlet of the column. Then the mobile phase was pumped through the head of the column while the column was rotated at a given rate. The eluate through the outlet of the column was continuously monitored with an LKB Uvicord III at 280 nm. Separations were performed under a wide range of operational conditions, of rotational speeds (0–80 rpm) and flow-rates (120 and 240 ml/h), while both upper aqueous and lower non-aqueous phases were tested as the stationary phase.

*Preparative counter-current chromatography with a long column*

The preparative capability of the present scheme was examined with a long column consisting of 10 coils with 2.5 cm core O.D. connected in series (tail–head connection). The column consisted of nearly 500 helical turns with a total capacity of approximately 900 ml. It was symmetrically mounted on the outer positions of the column holder. The solvent system and the samples were the same as those used in the partition efficiency studies. The sample solution was prepared by dissolving 500 mg of each DNP amino acid for a total of 1 g in 30 ml of the solvent consisting of equal amounts of the upper and lower phases. In each separation, the column was first filled with the stationary phase followed by sample injection through the sample port. Then the mobile phase was pumped through the head of the column at a rate of 120 ml/h while the column was rotated at the optimum rate determined by the preliminary studies. The eluates were collected with an LKB fraction collector to obtain a 12-ml fraction in each test tube. A 20-$\mu$l volume of each fraction was mixed with 3 ml of methanol to measure the absorbance at 430 nm with a Beckman DU spectrophotometer.

## RESULTS AND DISCUSSION

The typical results of the retention studies are illustrated in Fig. 2 where the percentage retention of the stationary phase relative to the total column capacity is plotted against the rotational speed of the column. The several lines drawn in each diagram indicate the effect of different flow-rates. Retention near 50% is considered to be ideal while lower levels of retention can be suitable for separations if the inclination of the curve is near horizontal which insures a stable retention of the stationary phase upon fluctuation of the rotational speed.

Fig. 2 shows retention in the large coil (2.5 cm O.D. core) mounted in the outer position of the column holder for the lower non-aqueous phase (A) and the upper aqueous phase (B). As is clearly observed, the retention levels of the non-aqueous phase are substantially lower than those of the aqueous phase throughout the applied rotational speeds. This higher level of retention produced by the aqueous phase may be largely attributed to its greater affinity to the glass wall of the column. The effects of the flow-rate on the retention of the stationary phase are also clearly shown in these diagrams; the slower the flow, the higher the retention levels. Figs. 2C and D similarly

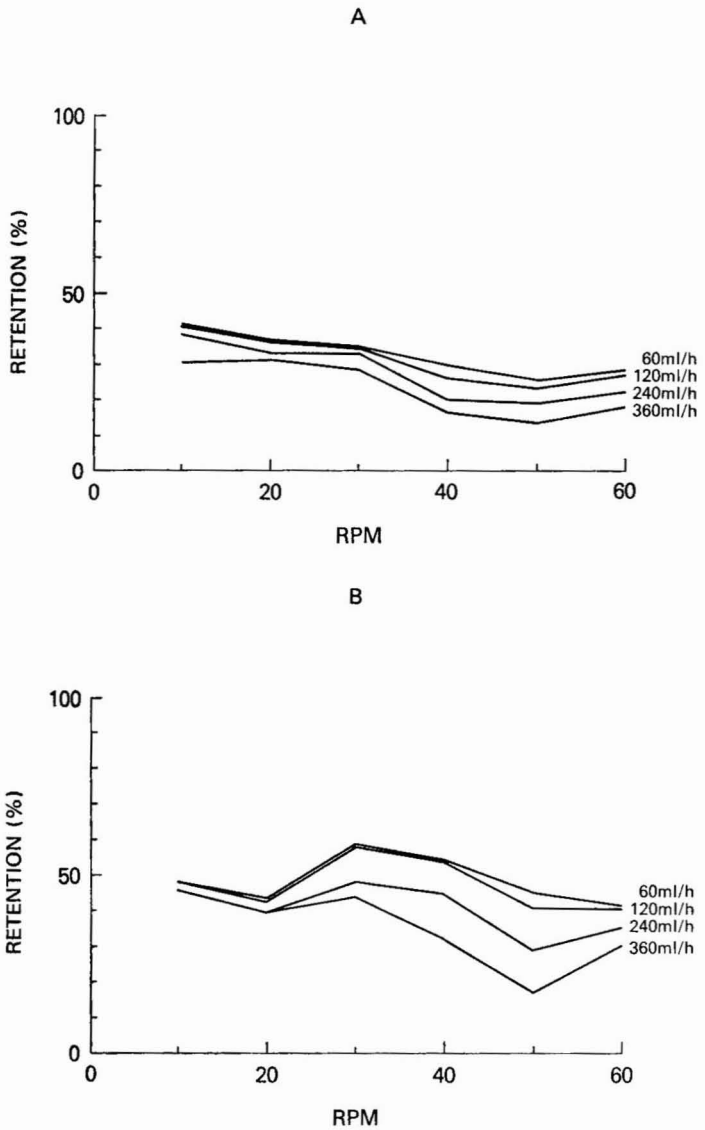


Fig. 2.

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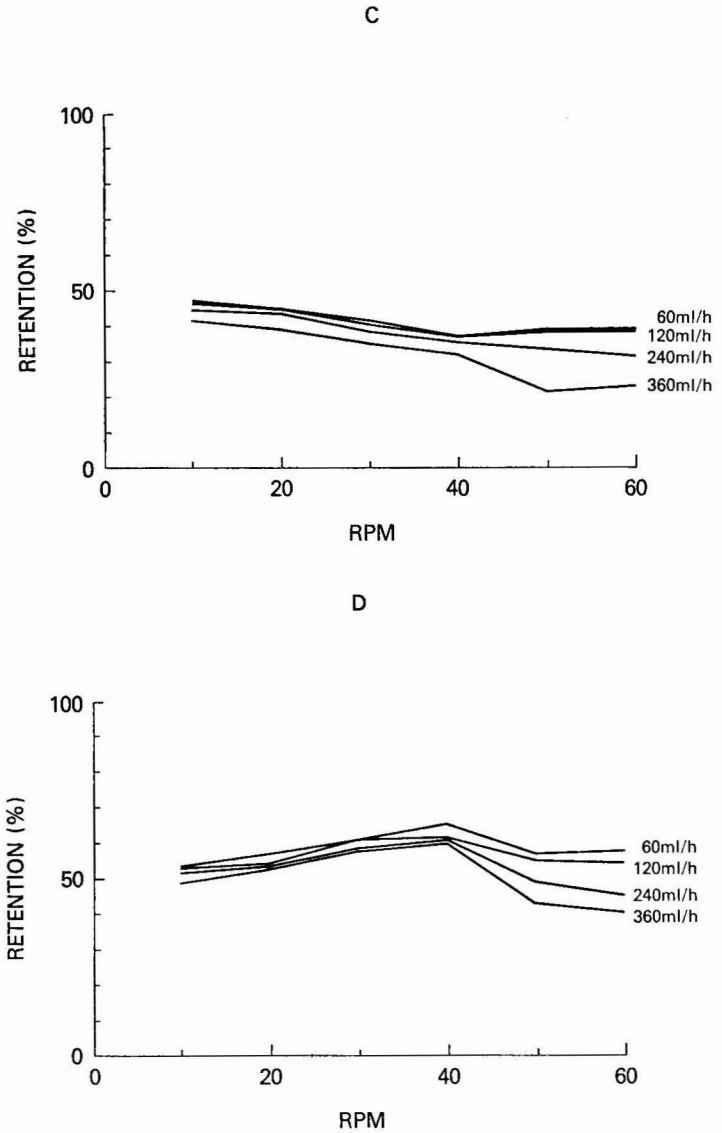


Fig. 2. The effect of rotational speed and flow-rate on stationary phase. Large coil in outside position: A, non-aqueous stationary phase; B, aqueous stationary phase. Small coil in outside position: C, non-aqueous stationary phase; D, aqueous stationary phase.

show the retention levels in the small coil (1.25 cm O.D. core) mounted in the outer position of the column holder for both stationary phases. The data clearly show that the retention levels produced by the small coil is substantially higher than those by the large coil for both non-aqueous and aqueous stationary phases. This may indicate that in the small core coil the linear velocity relative to the gravity becomes smaller resulting in less violent mixing of the two phases and, therefore, higher levels of phase retention at a given rotational speed occur.

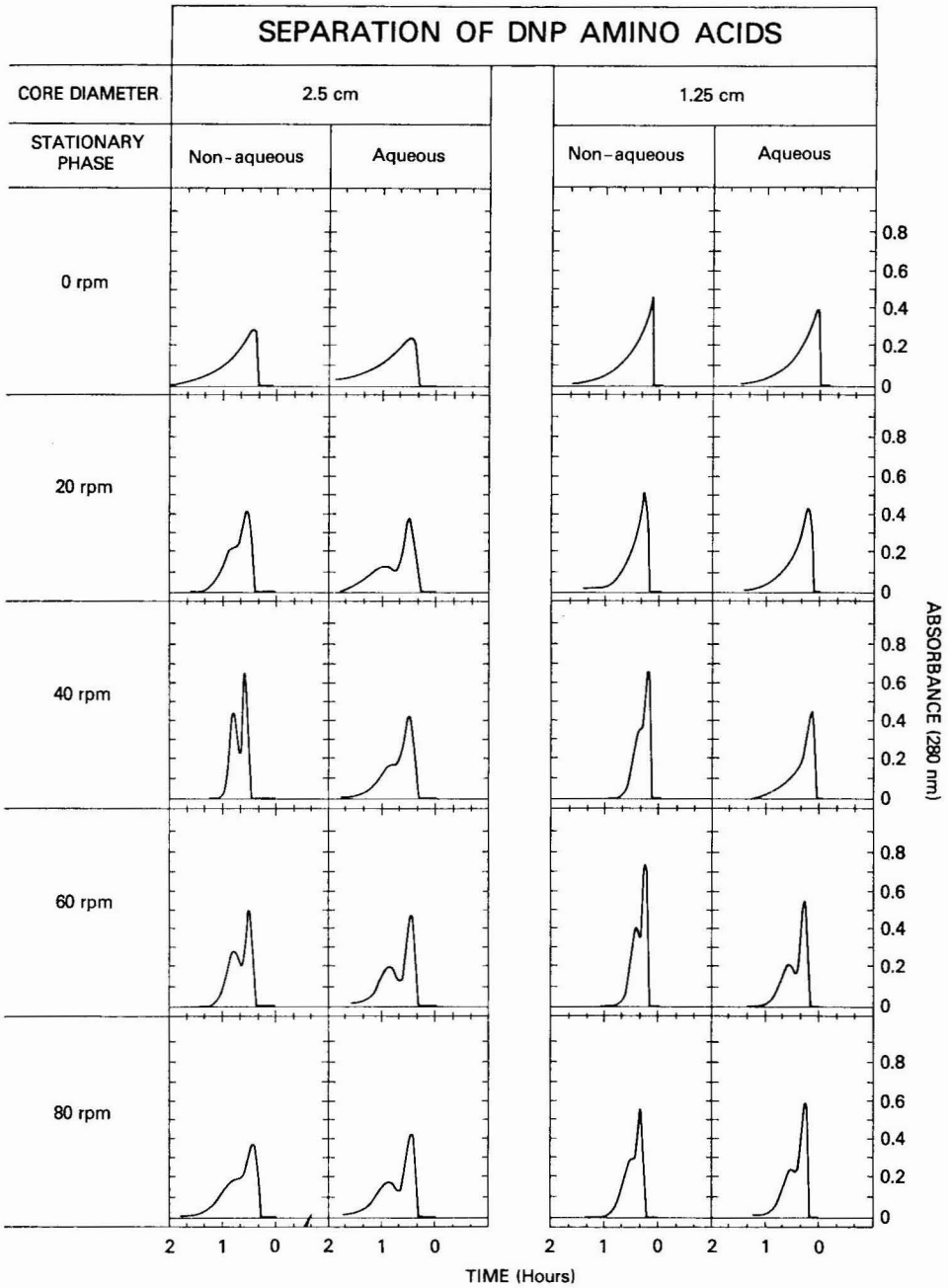


Fig. 3. The effect of rotational speed on the partition efficiency in the separation of DNP amino acids.

Data obtained with the same columns mounted in the inner position of the column holder gave similar results. Overall results indicate that satisfactory retention levels can be obtained with either type of coils under a wide range of rotational speeds and flow-rates.

Fig. 3. summarizes the results of DNP amino acid separation with a single coil mounted in the outer position of the column holder under a flow-rate of 120 ml/h. In each diagram, partition efficiency can be easily estimated by the resolution of the two peaks. In all groups the efficiency sharply increases as the rotational speed increases from 0 to 40–60 rpm, where the peak resolution becomes maximum. Further increase of the rotational speed to 80 rpm results in the loss of peak resolution. The optimum rotational speed thus ranges from 40 to 60 rpm for all groups. The highest peak resolu-

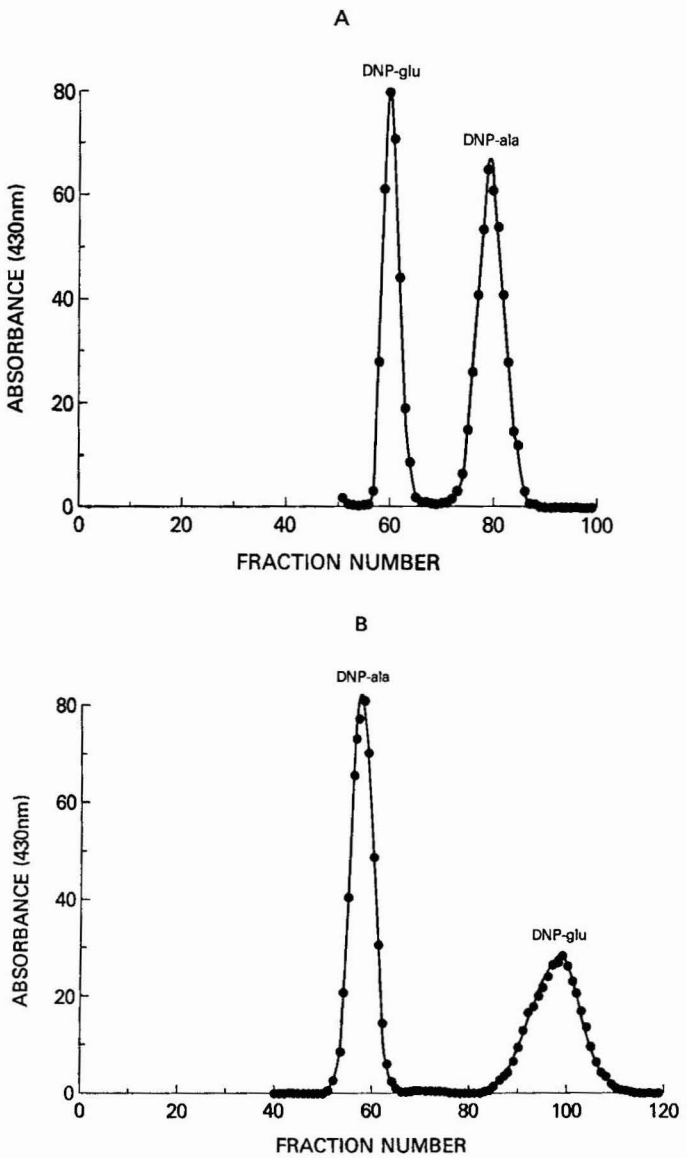


Fig. 4. Preparative-scale separations of DNP amino acids with a long coiled column. A, non-aqueous stationary phase; B, aqueous stationary phase.

tion is given by the large coil while the small coil could yield much higher resolution if two coils are connected to make the capacity equal to that of the single large coil. The results obtained with a higher flow-rate of 240 ml/h yielded less efficient separations in both small and large coils compared with those produced at 120 ml/h. The results obtained from the coils mounted in the inner position of the holder gave separations similar to those produced by respective coils mounted in the outer position of the column holder.

The preparative capability of the present counter-current chromatographic scheme was demonstrated by the separations of 1-g samples with a long column consisting of 10 large coils connected in series in the outside position. The separations were performed at a 120 ml/h flow-rate using both non-aqueous and aqueous stationary phases. Fig. 4A shows a chromatogram obtained at the optimum rotational speed of 40 rpm by using the non-aqueous phase as the stationary phase. The two DNP amino acids were completely resolved as symmetrical peaks and eluted out within 9 h. The partition efficiency calculated according to the standard formula[14] gives 1250 theoretical plates (T.P.) for the first peak and 880 T.P. for the second peak. Fig. 4B shows a similar chromatogram obtained at 60 rpm using the aqueous phase as the stationary phase. Because of higher aqueous phase retention, the peak resolution is much greater than that of the separation using the non-aqueous phase as stationary. The partition efficiency in the latter separation gives lower figures of 1000 T.P. for the first peak and 830 T.P. for the second peak.

The present scheme enables preparative-scale separation with a simple, compact apparatus. Separations are performed without the presence of solid supports and, therefore, complications such as sample loss, contamination and tailing of the solute peaks are minimized. The scheme yields high partition efficiency comparable to liquid chromatography while retaining high reproducibility and predictability inherent in the Craig counter-current distribution method. Because of its simplicity and mechanical stability, the present scheme can be further scaled up for large-scale industrial separations.

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# PEAK MOMENTS FOR GAS CHROMATOGRAPHIC COLUMNS WITH A PRESSURE DROP

O. PAZDERNÍK* and P. SCHNEIDER

*Institute of Chemical Process Fundamentals, Czechoslovak Academy of Sciences, 165 02 Prague 6 (Czechoslovakia)*

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## SUMMARY

Relationships have been derived for the first absolute and the second and third central moments of the chromatographic curve from a non-isobaric column for the Kubín and Kučera model (axial dispersion, external diffusion, internal diffusion, rate of adsorption). The dependence of the axial dispersion coefficient, mass transfer coefficient and effective diffusion coefficient on the pressure or carrier gas velocity is taken into account. In expressing the internal diffusion, the transition region between Knudsen and bulk diffusion is considered. By using the relationships for the moments, the dependence of the plate height on the carrier gas velocity is expressed (a modified Van Deemter equation for the non-isobaric case). If the rate of adsorption is not significant and internal diffusion takes place in the bulk region, it is possible to use the isobaric form of the Van Deemter equation with a corrected plate height.

## INTRODUCTION

The driving force for the flow of a carrier gas through a packed chromatographic column is the pressure drop, $\Delta p$ ($\Delta p = p_0 - p_e$, where $p_0$ and $p_e$ are the column inlet and outlet pressures, respectively). Consequently, the pressure $p(z)$ decreases and the interstitial linear velocity $v(z)$ increases along the column since because of the constancy of carrier gas mass flux, the following relationship holds* (on the assumption of ideal behaviour of the carrier gas):

$$p(z)\, v(z) = p_e v_e = \text{constant} \tag{1}$$

Recently, gas chromatography has been increasingly employed to determine the physico-chemical and chemical engineering parameters of rate processes taking place in a packed column. Therefore, it is necessary to include into the relevant relationships also the effect of varying linear velocity and pressure of the carrier gas

* The change in total mass flux due to the injected substance is considered to be negligible.

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because a number of parameters depend on these quantities (axial dispersion, external diffusion, internal diffusion).

So far attention has been paid, from this point of view, to some simpler models of processes in chromatographic columns (see, *e.g.*, refs. 1–4) or a solution has been obtained by using simplifying assumptions[5]. For the most general model of gas chromatography (the Kubín and Kučera model[6,7]), the expression for the first absolute moment of the outlet chromatographic curve has recently been obtained by Carleton *et al.*[8].

The aim of this work is to express the first absolute ($\mu_1'$) and the second and third central moments ($\mu_2$, $\mu_3$) of chromatographic curves in a non-isobaric column for the Kubín and Kučera model.

## THEORETICAL

### *Pressure and velocity profiles*

Even at relatively high carrier gas velocities, the Reynolds number in a packed column is usually low so that the Darcy equation is sufficient for the description of the carrier gas flow:

$$N = B^*(p/\mu)(1/R_gT)(-\mathrm{d}p/\mathrm{d}z) \tag{2}$$

where $N$ is the molar density of the carrier gas flow, $R_g$ the gas constant, $T$ the absolute temperature, $\mu$ the viscosity of the carrier gas, $B^*$ a constant characteristic of the packed column and $z$ the length coordinate of the column ($z = 0$ at the inlet, $z = L$ at the outlet). On integrating this equation, we obtain the following expression for the dependence of pressure and linear velocity on the position in the column:

$$p(z)/p_e = v_e/v(z) = \left[1 + (2\mu L\alpha v_e/B^* p_e)\left(1 - \frac{z}{L}\right)\right]^{1/2} \tag{3}$$

or

$$p(z)/p_e = v_e/v(z) = \left[1 + (P^2 - 1)\left(1 - \frac{z}{L}\right)\right]^{1/2} \tag{4}$$

where the subscript $e$ denotes the values at the column outlet and $P$ is the relative pressure at the column inlet ($P = p_0/p_e$).

### *Non-isobaric column*

If we divide the chromatographic column into differential segments of length $\mathrm{d}z$, the pressure and velocity of the carrier gas can be considered to be constant in each segment. The contributions to moments due to a differential segment $\mathrm{d}z$ are therefore the same as in an isobaric column in which the velocity and pressure are $v(z)$ and $p(z)$, respectively, and in which an identical shape of the input signal is used. Therefore,

$$\mu_n'(L) - \mu_n'(0) = \int_0^L \frac{\mathrm{d}}{\mathrm{d}z}(\mu_n')_{\mathrm{isobar}}\,\mathrm{d}z \quad (n = 0, 1, 2, \ldots) \tag{5}$$

$$\mu_n(L) - \mu_n(0) = \int_0^L \frac{\mathrm{d}}{\mathrm{d}z}(\mu_n)_{\mathrm{isobar}}\,\mathrm{d}z \quad (n = 0, 1, 2, \ldots) \tag{6}$$

where $\mu_n'$ and $\mu_n$ denote the $n$th absolute and central moment, respectively, of the chromatographic curve at the position $z$[$\mu_n'(L)$ and $\mu_n(L)$ are the moments of the outlet chromatographic curve at $z = L$] and the subscript isobar denotes the moments for a column with a negligible pressure drop.

The moments $\mu_n'$ and $\mu_n$ for the isobaric and non-isobaric cases are defined in the usual way as

$$\mu_n'(z) = \int_0^\infty t^n c(z,t)\mathrm{d}t / \int_0^\infty c(z,t)\mathrm{d}t \quad (n = 0, 1, 2, \ldots) \tag{7}$$

$$\mu_n(z) = \int_0^\infty (t - \mu_1')^n c(z,t)\mathrm{d}t / \int_0^\infty c(z,t)\mathrm{d}t \quad (n = 0, 1, 2, \ldots) \tag{8}$$

where $c(z,t)$ is the time dependence of the concentration of an injected substance at the position $z$.

Regardless of the pressure conditions in the column, for an input signal in the shape of a rectangular pulse of width $t_0$ it holds that

$$\mu_1'(0) = t_0/2;\ \mu_2(0) = t_0^2/12;\ \mu_3(0) = 0 \tag{9}$$

If we use an input signal in the form of a Dirac function, all of the input moments are zero:

$$\mu_n'(0) = \mu_n(0) = 0 \quad (n = 1, 2, \ldots) \tag{10}$$

*Kubín and Kučera isobaric model*

In the Kubín and Kučera model, the processes in a chromatographic column can be described by the following mass balances of the injected substance[6,7,9,10]:
column

$$E(\partial^2 c/\partial z^2) - v(\partial c/\partial z) - (\partial c/\partial t) - (3\gamma/\beta)(D/R)(\partial q/\partial r\,|_R) = 0 \tag{11}$$

particles of the column packing:

$$D[(\partial^2 q/\partial r^2) + (2/r)(\partial q/\partial r)] - \beta(\partial q/\partial t) - \varrho_p(\partial w/\partial t) = 0 \tag{12}$$

Because of the low concentration, a linear rate equation is assumed for the rate of adsorption of the injected substance:

$$\partial w/\partial t = k_d[(K\beta/\varrho_p)q - w] \tag{13}$$

If the adsorption is in equilibrium, this equation turns into the linear (Henry) adsorption isotherm.

The partial differential eqns. 11 and 12 are supplemented by the following boundary and initial conditions:

$$D(\partial q/\partial r|_R) = k_c[c - q(R)] \tag{14}$$

$$r = 0 \quad \partial q/\partial r = 0 \tag{15}$$

$$t \leqslant 0 \quad c = q = w = 0 \tag{16}$$

$$z = 0 \quad t > 0 \quad c = c_0(t) \tag{17}$$

In eqns. 11–17, $c$ and $q$ denote the molar concentrations of the injected substance in the carrier gas in the space between the particles of the packing and in the pores of these particles, respectively, $w$ is the molar amount of injected substance adsorbed per unit mass of packing particles, $r$ is the length coordinate of the spherical particles of the packing ($r = 0$ at the centre, $r = R$ at the external surface), $t$ is the time from the beginning of input signal, $E$ is the axial dispersion coefficient, which is usually expressed as

$$E = (\mathscr{D}/\tau) + \varkappa Rv \tag{18}$$

$\mathscr{D}$ is the binary bulk diffusion coefficient of the injected substance–carrier gas pair, $\tau$ is the tortuosity of the space between the particles of the packing, $\varkappa$ is a numerical coefficient characterizing the contribution of turbulent diffusion to axial dispersion, $R$ is the radius of the particles of the packing, $k_d$ and $K$ are the rate constant of desorption and the dimensionless equilibrium constant of adsorption of the injected substance on the internal surface of the particles of the packing, respectively, $k_c$ is the mass transfer coefficient of the injected substance between the bulk of the carrier gas and the external surface of the particle, $D$ is the effective diffusion coefficient in the packing particle, $\alpha$ is the external porosity (void volume between particles per unit column volume), $\beta$ is the internal porosity (pore volume in a particle per unit of its volume) and $\gamma$ is the ratio of the void volume in the particle (pores) to that between particles [$\gamma = (1 - \alpha)\beta/\alpha$].

By solving the system of eqns. 11–17 by Laplace transformation, it is possible to obtain the following expressions for moments[6,7,9,11,12]:

$$[\mu_1'(z)]_{\text{isobar}} = \mu_1'(0) + (z/v)\,(1 + \delta_0) \tag{19}$$

$$[\mu_2(z)]_{\text{isobar}} = \mu_2(0) + (2z/v)\,[\delta_1 + (E/v^2)\,(1 + \delta_0)^2] \tag{20}$$

$$[\mu_3(z)]_{\text{isobar}} = \mu_3(0) + (6z/v)\,[\delta_2 + 2(E/v^2)\delta_1\,(1 + \delta_0) + 2(E/v^2)^2\,(1 + \delta_0)^3] \tag{21}$$

where

$$\delta_0 = \gamma(1 + K) \tag{22}$$

$$\delta_1 = \delta_a + \delta_f + \delta_d \tag{23}$$

$$\delta_a = (\delta_0^2/\gamma)\,(K/k_d)/(1 + K)^2 = \gamma K/k_d \tag{24}$$

$$\delta_f = (\delta_0^2/\gamma)\,(R\beta/3k_c) \tag{25}$$

$$\delta_d = (\delta_0^2/\gamma)\,(R^2\beta/15\,D) \tag{26}$$

$$\delta_2 = [\delta_1^2 + (3/7)\,\delta_d^2 + (\delta_a^2/K)]/\delta_0 \tag{27}$$

*Non-isobaric model*

Under non-isobaric conditions it is necessary to take into account the change in the carrier gas velocity along the column and, simultaneously, the corresponding changes in transport parameters which depend on the carrier gas velocity or pressure.

Using eqn. 18 for the description of axial dispersion, then, with respect to eqn. 1 and to the fact that $\mathscr{D} \approx 1/p$, it holds that

$$E/E_e = v/v_e = p_e/p \tag{28}$$

The mass transfer coefficient, $k_c$, is usually correlated in the form of the Sherwood number with the Schmidt and Reynolds numbers, *i.e.*, as $Sh = f(Re,Sc)$. Here neither $Re$ nor $Sc$ depends on the position in the column [$Sc = (Sc)_e$, $Re = (Re)_e$], so that $Sh = (Sh)_e$ and hence

$$k_c/(k_c)_e = v/v_e = p_e/p \tag{29}$$

With internal diffusion the situation is more complicated. If diffusion takes place in the Knudsen region the effective diffusion coefficient does not change along the column and $D = D_e$. If diffusion occurs in the bulk region then $D/D_e = v/v_e = p_e/p$. For the transition between these regions it is therefore possible to write approximately

$$D/D_e = (v/v_e)^m = (p_e/p)^m \tag{30}$$

where $m$ takes values between 0 and 1 ($m = 0$ in the Knudsen region, $m = 1$ for the bulk diffusion). With the exception of porous particles containing only very narrow pores in which the Knudsen diffusion occurs (see, *e.g.*, ref. 9), it can be expected that the diffusion transport will take place mostly in wider transport pores (see, *e.g.*, refs. 10 and 13) close to the bulk region.

The parameters describing the rate and equilibrium of adsorption of an injected substance ($k_d$, $K$) do not depend on the pressure and velocity of the carrier gas.

*Non-isobaric moments*

By integrating according to eqns. 5 and 6 on using relations 19–21 and dependences 28–30, it is possible to obtain the following relationships for the moments of the outlet chromatographic curve from a non-isobaric column:

$$\mu_1'(L) = \mu_1'(0) + (L/v_e) f_1 (1 + \delta_0) \tag{31}$$

$$\mu_2(L) = \mu_2(0) + (2L/v_e) [f_1\delta_a + f_2(\delta_f)_e + f_{m+1} (\delta_d)_e + f_2 (E_e/v_e^2) (1 + \delta_0)^2] \tag{32}$$

$$\begin{aligned}\mu_3(L) = \mu_3(0) + (6L/v_e) \{&(1/\delta_0)[f_1\delta_a^2 (1 + K)/K + f_3 (\delta_f)_e{}^2 + (10/7) f_{2m+1} (\delta_d)_e^2 + \\ &+ 2\delta_a (f_2 (\delta_f)_e + f_{m+1} (\delta_d)_e) + 2f_{m+2} (\delta_f)_e (\delta_d)_e] + 2(E_e/v_e^2) (1 + \delta_0) [f_2\delta_a + \\ &+ f_{m+2} (\delta_d)_e + f_3 (\delta_f)_e] + 2f_3 (E_e/v_e^2)^2 (1 + \delta_0)^3\}\end{aligned} \tag{33}$$

where $\delta_0$ and $\delta_a$ are given by eqns. 22 and 24 and

$$(\delta_f)_e = (\delta_0^2/\gamma) [R\beta/3(k_c)_e] \tag{34}$$

$$(\delta_d)_e = (\delta_0^2/\gamma) (R^2\beta/15\, D_e) \tag{35}$$

The correction factors $f_k (k = 1\text{–}3)$ can be expressed as functions of the relative inlet pressure, $P$:

$$f_k = \int_0^1 (v_e/v)^k \, \mathrm{d}(z/L) = [2/(k+2)]\,(P^{k+2} - 1)/(P^2 - 1) \tag{36}$$

The dependences $f_k$ *vs.* $P$ are illustrated in Fig. 1; it can be seen that the correction factors can take comparatively high values. It can also be seen that for lower $P$ holds $f_k(P) \approx [f_1(P)]^k$.

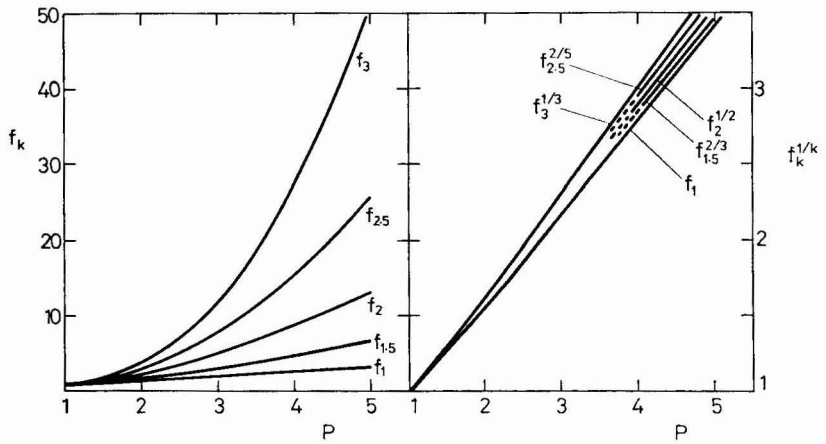


Fig. 1. Correction factors for moments.

*First absolute moment.* The factor $f_1$ in eqn. 31 for $\mu_1'(L)$ is identical with the James–Martin correction for retention times[14,15] and has recently been derived by Carleton *et al.*[8] for the Kubín and Kučera model by solving a system of balance equations for a non-isobaric column. On introducing the corrected linear volocity $v_{corr}$, as

$$v_{corr} = v_e/f_1 \tag{37}$$

the dependence $[\mu_1'(L) - \mu_1'(0)]$ *vs.* $L/v_{corr}$ must be linear and pass through the origin as in the isobaric case (*cf.*, eqn. 19). From the slope of this dependence it is possible to determine the adsorption parameter, $\delta_0$, or the equilibrium adsorption constant of injected substance, $K$.

From a comparison of first absolute moments in the non-isobaric ($P > 1$) and isobaric ($P = 1$) cases at the same outlet velocity, $v_e$, it follows that $\mu_1'(L)$ in the non-isobaric case is always higher because $f_1 > 1$. This is a consequence of lower linear carrier gas velocities in the upstream parts of column in comparison with $v_e$.

*Second central moment.* Under otherwise identical conditions, at the same velocity $v_e$, the second central moment in the non-isobaric column is always higher than in the isobaric case (corrections $f_1, f_2, f_{m+1} > 1$). The existence of a pressure drop consequently contributes to the peak spreading. The relative increase in the contributions of axial dispersion and external diffusion is the same; the increase in the internal diffusion contribution depends on the region in which internal diffusion takes place (parameter $m$). With bulk diffusion ($m = 1$), this increase is the same as for

axial dispersion and external diffusion. If the Knudsen diffusion is significant ($m < 1$) the contribution of internal diffusion decreases.

If adsorption is very rapid ($\delta_a \to 0$) and internal diffusion is of the bulk type ($m = 1$), the relative increase in the second central moment due to the pressure drop in the column is $f_2 - 1$. Consequently, at P = 1.1, 1.5 and 2 it represents 10.5%, 62.5% and 150%, respectively.

*Third central moment.* The third central moment, $\mu_3(L)$, characterizes the asymmetry of the outlet chromatographic curve. It is evident from eqn. 33 and from the values of the correction factors in Fig. 1 that the pressure drop increases the asymmetry considerably. For instance, for $\delta_a \to 0$ and internal diffusion in the bulk region ($m = 1$), the relative increase in the third moment compared with the isobaric value is $f_3 - 1$. Thus, for $P = 1$, 1.5 and 2 it amounts to 16.3%, 111% and 313%, respectively. The increase in contributions to the third moment due to the external and internal diffusion and axial dispersion is the same as with the second central moment.

*Plate height.* When evaluating the parameters of processes taking place in a chromatographic column, the plate height, $H$, is often used. It is defined as

$$H = L[\mu_2(L) - \mu_2(0)]/[\mu_1'(L) - \mu_1'(0)]^2 \tag{38}$$

The dependence of $H$ on the carrier gas velocity, $v_e$, can easily be obtained even in the non-isobaric case by combining eqns. 38 and 18 and 31 and 32 in the form

$$H = Ag_2 + (Bg_2/v_e) + Cv_e \tag{39}$$

where

$$A = 2\varkappa R \tag{40}$$

$$B = 2\,\mathscr{D}_e/\tau \tag{41}$$

$$C = C_a g_1 + C_f g_2 + C_d g_{m+1} \tag{42}$$

$$C_a = 2\,\delta_a/(1 + \delta_0)^2 \tag{43}$$

$$C_f = 2\,(\delta_f)_e/(1 + \delta_0)^2 \tag{44}$$

$$C_d = 2\,(\delta_d)_e/(1 + \delta_0)^2 \tag{45}$$

and the corrections $g_k$ ($k$ = 1–2) are defined as

$$g_k = f_k/f_1 = [9/(2k + 4)]\,(P^2 - 1)\,(P^{k+2} - 1)/(P^3 - 1)^2 \tag{46}$$

The dependence $g_k(P)$ is illustrated in Fig. 2*. Eqn. 39 is related to the modified Van Deemter equation, into which it turns in the isobaric case ($P = 1$, *i.e.*, $g_1 = g_2 = g_{m+1} = 1$).

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* The slight dependence of $g_2$ on $P$ is a consequence of the approximate validity of the relationship $f_2 \approx f_1^2$ ($g_2 = f_2/f_1^2 \approx 1$).

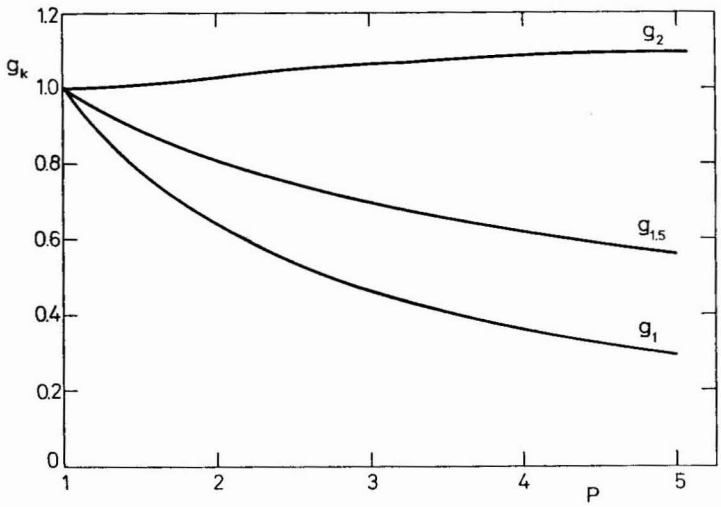


Fig. 2. Correction factors for the Van Deemter equation (eqn. 38).

The contributions to constant $C$ (eqn. 42) change in the non-isobaric case in a different way. In comparison with the isobaric case, the contribution of the rate of adsorption diminishes ($C_a g_1$; $g_1 < 1$), as the contribution of internal diffusion diminishes in the case when the Knudsen diffusion plays a more significant role ($C_d g_{m+1}$; *e.g.*, for $m = 0.5$ $g_{1.5} < 1$). The contribution of external diffusion ($C_f g_2$) is influenced only slightly by the change in the pressure drop over the column (*e.g.*, for $P = 5$, $g_2 = 1.095$); the same holds for internal diffusion in the bulk region ($C_d g_2$). Likewise the terms characterizing the turbulent ($A g_2$) and the diffusion ($B g_2$) parts of axial dispersion change slightly.

It often occurs that the rate of adsorption plays a negligible role ($C_a \to 0$) and the internal diffusion in close to the bulk region ($m \to 1$). Then it is possible to rewrite eqn. 39 in the form

$$H/g_2 = A + (B/v_e) + (C_d + C_f)v_e \tag{47}$$

The corrected plate height, $H/g_2$, therefore exhibits a dependence on velocity, $v_e$, identical with that for isobaric conditions. Since for $P < 5$ the correction function $g_2$ differs from 1 at most by 10%, it is possible to use, in a rougher approximation, the uncorrected values of $H$ on the left-hand side of eqn. 47.

## CONCLUSION

Using the relationships for moments of outlet chromatographic curves in non-isobaric columns, it is possible to estimate the effect of pressure drop on the separation efficiency of the column: the retention times [$\sim\mu_1'(L)$] of components of the separated mixture increase in the same way (factor $f_1$); however, the peaks are simultaneously spread [$\sim\mu_2(L)$] and their asymmetry is increased [$\sim\mu_3(L)$]. Consequently, partial overlapping of peaks takes place and the quality of separation is reduced.

The presence of pressure drop does not cause any problems in chromatographic measurements aimed at evaluating the adsorption equilibrium constant of

an injected substance ($K$ in the parameter $\delta_0$, eqn. 22). Provided that the corrected carrier gas velocity, $v_{corr}$, (eqn. 37) is used, it is possible to evaluate the parameter $\delta_0$ from the dependence $[\mu_1'(L) - \mu_1'(0)]$ *vs.* $L/v_{corr}$ in the same way as under isobaric conditions.

A more complicated situation occurs when it is necessary to evaluate transport parameters by using higher moments of outlet chromatographic curves or plate heights. Then we usually start from the measurements at a number of linear velocities and/or particle sizes of the packing and a graphical procedure or numerical fitting of experimentally determined moments is used. Eqns. 32, 33 and 39 contain, in addition to constant parameters, the correction functions $f_k$ and $g_k$ (eqns. 36 and 46), which are expressed through the easily measurable inlet pressure. Then it is necessary to express P in eqns. 36 and 46 from the relationship

$$P = [1 + (2\mu L\alpha\, v_e/B^* p_e)]^{1/2} \tag{48}$$

which follows from eqns. 2 and 3. Further, it is necessary to express the dependence of $E_e$ on $v_e$, *e.g.*, by using eqn. 18*, and the dependence of $(k_c)_e$ on $v_e$ by employing the chemical engineering correlations for packed beds. As external diffusion usually represents a negligible resistance, the last step is not decisive.

Under the usual conditions, adsorption is in the vicinity of equilibrium ($\delta_a \to 0$), external diffusion plays only a negligible role and transport in the packing particles takes place mostly in wide transport pores ($m \to 1$). Then it is possible to evaluate easily the effective diffusion coefficient, $D_e$, from the slope of linear asymptote of the dependence of $H/g_2$ *vs.* $v_e$ for higher linear velocities of the carrier gas ($C_d$, eqn. 45). For the usual pressure drops the correction $g_2$ can also be omitted and the part of the dependence $H$ *vs.* $v_e$ for higher velocities can be used directly.

The validity of eqns. 5 and 6 is substantiated by the additivity of moments. Further proof follows from the identity of correction functions $f_1$ and $f_2$ obtained for the case of a linear pressure decrease along the column with corrections obtained via the exact solution of the non-isobaric column material balance (partial differential) equations; for this simple pressure profile this can be easily done in a manner similar to that proposed by Carleton *et al.*[8].

## SYMBOLS

| | |
|---|---|
| $A$, $B$, $C$ | term of the Van Deemter equation (cm, cm²/sec, sec) |
| $B^*$ | constant characteristic of the packed column (cm²/sec) |
| $c(z,t)$ | time dependence of the concentration of the injected substance at position $z$ in the interstitial volume (mol/cm³) |
| $C_a$,$C_d$,$C_f$ | contributions to the constant $C$ in the Van Deemter equation (sec) |
| $\mathscr{D}$ | binary bulk diffusion coefficient (cm²/sec) |
| $D$ | effective diffusion coefficient of injected substance in particles of the packing (cm²/sec) |
| $E$ | axial dispersion coefficient (cm²/sec) |

* This dependence is already incorporated in eqn. 39.

| | |
|---|---|
| $f_k$ | correction factor, eqn. 36 |
| $g_k$ | correction factor, eqn. 46 |
| $H$ | plate height (cm) |
| $k_c$ | mass transfer coefficient (cm/sec) |
| $k_d$ | desorption rate constant for the injected substance ($sec^{-1}$) |
| $K$ | adsorption equilibrium constant for the injected substance |
| $L$ | column length (cm) |
| $m$ | exponent, eqn. 30 |
| $N$ | molar density of the carrier gas flow ($mol/cm^2 \cdot sec$) |
| $p, p(z)$ | column pressure at position $z$ ($dyn/cm^2$) |
| $P$ | relative pressure at the column inlet ($P = p_0/p_e$) |
| $q$ | concentration of injected substance in pores of the particles of the packing ($mol/cm^3$) |
| $r$ | length coordinate in spherical packing particle (cm) |
| $R$ | radius of the particles of the packing (cm) |
| $R_g$ | gas constant ($erg/mol \cdot °K$) |
| $Re$ | Reynolds number, $Re = 2Rv\varrho/\mu$ |
| $Sc$ | Schmidt number, $Sc = \mu\varrho/\mathscr{D}$ |
| $Sh$ | Sherwood number, $Sh = 2Rk_c/\mathscr{D}$ |
| $t$ | time (sec) |
| $t_0$ | width of the input rectangular pulse (sec) |
| $T$ | absolute temperature (°K) |
| $v, v(z)$ | interstitial carrier gas velocity at position $z$ (cm/sec) |
| $w$ | molar amount of injected substance adsorbed per unit particle mass (mol/g) |
| $z$ | length coordinate of column; $z = 0$ at inlet (cm) |
| $\alpha$ | external porosity |
| $\beta$ | internal porosity |
| $\gamma$ | ratio of void volume in particle (pores) and between particles; $\gamma = (1 - \alpha)\beta/\alpha$ |
| $\delta_0$ | contribution to moments |
| $\delta_a, \delta_d, \delta_f, \delta_1$ | contributions to moments (sec) |
| $\delta_2$ | contribution to moments ($sec^2$) |
| $\varkappa$ | numerical coefficient (eqn. 18) |
| $\mu$ | carrier gas viscosity ($g/cm \cdot sec$) |
| $\mu_n'(z)$ | $n$th absolute moment at position $z$ ($sec^n$) |
| $\mu_n(z)$ | $n$th central moment at position $z$ ($sec^n$) |
| $\varrho$ | carrier gas density ($g/cm^3$) |
| $\varrho_p$ | apparent packing density ($g/cm^3$) |
| $\tau$ | tortuosity of the interparticle space |

*Subscripts*

| | |
|---|---|
| 0 | column inlet |
| $e$ | column outlet |
| isobar | negligible pressure drop |
| $a$ | adsorption |
| $d$ | internal diffusion |
| $f$ | external diffusion |

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# DETERMINATION OF ACTIVITY COEFFICIENTS AT VERY LOW CONCENTRATIONS BY THE INERT GAS STRIPPING METHOD*

B. DOLEŽAL* and M. POPL

*Department of Instrumental Analysis, Institute of Chemical Technology, 166 28 Prague 6 (Czechoslovakia)*

and

R. HOLUB

*Department of Physical Chemistry, Institute of Chemical Technology, 166 28 Prague 6 (Czechoslovakia)*

## SUMMARY

Based on a theoretical concept of changes in solute concentration brought about by the passage of an inert gas, a measuring apparatus was set up and a saturation vessel devised that alters the equilibrium between the liquid and gaseous phases. The inert gas flow-rate was optimized. Two variants of the experimental procedure were tested on *n*-pentane and *n*-octane. The results obtained on passage of the pure inert gas were within the limits of error of the results obtained by employing pre-saturation.

The proposed method is not too laborious and is easy to perform; on the other hand, the period required for the necessary decrease in concentration is very long (up to several days).

The pre-saturation variant can be used when highly volatile solvents are involved, whereas the other variant has to be used if trace amounts of impurities in the solvent could affect the concentration of the solute in the solution being measured.

## INTRODUCTION

Activity coefficients of components at infinite dilution are important thermodynamic quantities used in the characterization of phase equilibria. There are a number of methods (in chromatography they are "static" methods) for the determination of activity coefficients of solutes in finite concentrations; their extrapolation to the concentration limits, however, is usually inaccurate or completely unsuccessful. A common procedure for the determination of the $\gamma^{\infty}$ values is the "retention time method" in gas–liquid chromatography (GLC), based on a thermodynamic characterization of the equilibrium between the solute and the solvent in a GLC column.

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Experimental and calculation correlations have been proposed for obtaining the true values of the coefficients from experimental data; however, it is not always possible to evaluate adequately the effect of adsorption on the gas–liquid or liquid–solid interface. From this point of view, the inert gas stripping method[1] appears to be suitable for the determination of the limiting activity coefficients for very low concentrations of the solute, because the solvent surface area to weight ratio is negligible compared with that in a GLC column.

## THEORETICAL

In accordance with the general procedure presented elsewhere[1,2], relationships between the results of chromatographic analysis of the solute and solvent and the volume of inert gas passed were derived[3], based on a thermodynamic description of the solute (1)–solvent(2)–inert gas(3) system.

*Balance of the components leaving the solution*

The relationship between the instantaneous solution composition and equilibrium gaseous phase can be expressed using Raoult's and Henry's laws (assuming $x_1 \to 0$, $x_2 \to 1$, $x_3 \to 0$):

$$f_1^- = x_1 \gamma_1 f_1^0 \tag{1}$$

$$f_2^- = x_2 f_2^0 \tag{2}$$

$$f_3^- = x_3 H_{3,2} \tag{3}$$

where $f_i^-$ ($i = 1,2,3$) is the fugacity of component $i$ in the system outlet, $f_i^0$ ($i = 1,2$) is the fugacity of the pure component at the system temperature, $\gamma_1$ is activity coefficient of the solute at a given composition and temperature of the liquid phase, $H_{3,2}$ is Henry's constant for inert in pure solvent,

$$x_i = \frac{n_i}{n_1 + n_2 + n_3} \tag{4}$$

($i = 1,2,3$), $x_i$ is the molar fraction of component $i$ in the liquid phase and $n_1$, $n_2$ and $n_3$ are moles of solute, solvent and dissolved inert gas, respectively, in the solution.

The system fugacity is given approximately as the sum of fugacities of the components according to eqn. 1, 2 and 3:

$$f^- \approx f_1^- + f_2^- + f_3^- \tag{5}$$

and then

$$\mathrm{d}n_1^- = x_1 \gamma_1 \cdot \frac{f_1^0}{f^-} \cdot \mathrm{d}n^- \tag{6}$$

$$\mathrm{d}n_2^- = x_2 \cdot \frac{f_2^0}{f^-} \cdot \mathrm{d}n^- \tag{7}$$

where $dn_1^-$ and $dn_2^-$ are infinitesimal masses of solute and solvent leaving the solution simultaneously with $dn_3^-$ moles of inert gas. Further,

$$dn^- = dn_1^- + dn_2^- + dn_3^- \tag{8}$$

From eqns. 6–8 the relationship

$$dn^- = \frac{f^- \, dn_3^-}{f^- - x_1 \gamma_1 f_1^0 - x_2 f_2^0} \tag{9}$$

can be obtained, which, in combination with eqns. 4 and 6 and eqns. 4 and 7 gives the explicit expression for the instantaneous solution composition and leaving gaseous phase:

$$dn_1^- = \frac{n_1}{n_2} \cdot \gamma_1 \cdot \frac{f_1^0}{f^-} \cdot \frac{dn_3^-}{A} \tag{10}$$

or

$$dn_2^- = \frac{f_1^0}{f^-} \cdot \frac{dn_3^-}{A} \tag{11}$$

where

$$A = 1 - \frac{f_2^0}{f^-} + \left(1 - \gamma_1 \cdot \frac{f_1^0}{f^-}\right) \frac{n_1}{n_2} + \frac{n_3}{n_2} \tag{12}$$

*Balance of the components entering the solution from the overall balance*

There are two main possibilities: either pure inert gas is used[1,2] or the inert gas is saturated with solvent vapour so that loss of the solvent is prevented. In view of the assumption of a highly dilute solution, most of the quantities in eqns. 10 and 11 can be regarded[3] as independent of concentration and their values approximated by those occurring at the beginning of the measurement. The factor $\gamma_1$ in eqn. 12 can be considered as a correlation factor, as the effect of all the terms except $1 - f_2^0/f^-$ on $A$ is virtually negligible.

*Passage of pure inert gas*

In this instance

$$dn_1 = -dn_1^- \tag{13}$$

$$dn_2 = -dn_2^- \tag{14}$$

$$dn_3^- = dn_3^+ - \frac{f_3^-}{H_{3,2}} \cdot dn_2 \tag{15}$$

where the superscript $+$ refers to entering the solution. Combining eqns. 10 and 11, we obtain

$$d\ln n_1(n_3^+) = \gamma_1 \cdot \frac{f_1^0}{f_2^0} \cdot d\ln n_2(n_3^+) \tag{16}$$

where $n_1(n_3^+)$ and $n_2(n_3^+)$ are functions of the masses of the inert gas that entered the solution during the experiment. The change in the mass of the solute can be obtained fairly accurately by measuring the areas, $S_1$, enclosed by the chromatographic elution peaks of the substance; the decrease in the mass of solvent has been described by Burnett[2]. With this arrangement, $\gamma_1$ can be expressed as

$$\gamma_1 = \frac{f_2^0}{f_1^0}\left\{1 + \frac{\mathrm{d}\ln S_1(n_3^+)/S_1(0)}{\mathrm{d}\ln\left[1 - \frac{1}{n_2(0)} \cdot \frac{f_2^0}{f^-} \cdot \frac{n_3^+}{A - f_2^0/f^- \cdot f_3^-/H_{3,2}}\right]}\right\} \tag{17}$$

where (0) denotes the quantity in question at the beginning of the experiment and $(n_3^+)$ denotes the value after the entry of $n_3^+$ moles of pure inert gas into the solution.

*Passage of inert gas saturated with solvent vapour*

The inert gas saturated with solvent vapour is fed into the solution under conditions such that changes in amount of solvent are prevented. There is only one significant difference in the conditions with pre-saturation and the use of a saturation vessel, namely the pressure. The effect of this difference is offset by a corresponding rise in temperature:

$$P_2^0(T^+) = \frac{x_2^-}{x_2^+} \cdot \frac{P^+}{P^-} \cdot P_2^0(T^-); P_2^0(T^+) = >T^+ \tag{18}$$

where + refers to the values in the pre-saturator and − to those in the saturation vessel.

The change in the mass of the solute (eqn. 10), can be found accurately by GLC analysis; the activity coefficient for very low concentration can be written as

$$\gamma_1 = -n_2(0) \cdot \frac{f^-}{f_1^0} \cdot A \cdot \frac{\mathrm{d}\ln S_1(n_3^+)}{\mathrm{d}n_3^+} \tag{19}$$

## EXPERIMENTAL

*n*-Pentane was obtained from Carlo Erba (Milan, Italy), *n*-octane from VEB Laborchemie (Apolda, G.D.R.) and carbon tetrachloride from Lachema (Brno, Czechoslovakia).

The two variants of the method were tested on the *n*-pentane(1)–*n*-octane(2) system using nitrogen(3) as the inert gas. Each experiment consisted of 20–30 analyses, in which the amount of inert gas passed was read, two or three injections were made in rapid succession and the $n_3^+$ value was read again and averaged with the preceding one. This sequence was performed approximately ten times. The concentration dependence of $\gamma_{CCl_4}$ in $C_8$ was obtained based on analogous procedures.

The experimental data required for the determination of $\gamma^\infty$ based on eqn. 17 or 19 were measured on the apparatus depicted in Fig. 1. Its basic unit was the saturation vessel (5), depicted in Fig. 2.

The volume of solution measured was approximately 130–140 ml. The coil

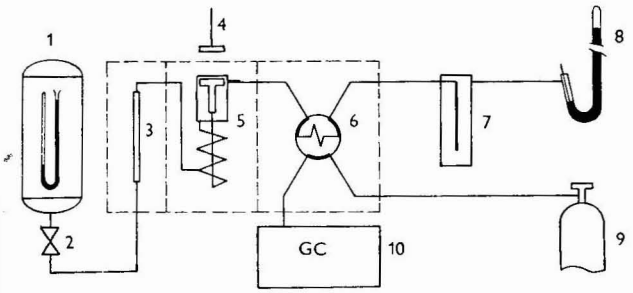


Fig. 1. Apparatus. 1 = Pressure vessel containing the inert gas; 2 = gas flow control; 3 = pre-saturator; 4 = rotating permanent magnet; 5 = saturation vessel; 6 = proportioning valve; 7 = capacity vessel; 8 = manostat; 9 = pressure vessel containing the carrier gas; 10 = gas chromatograph.

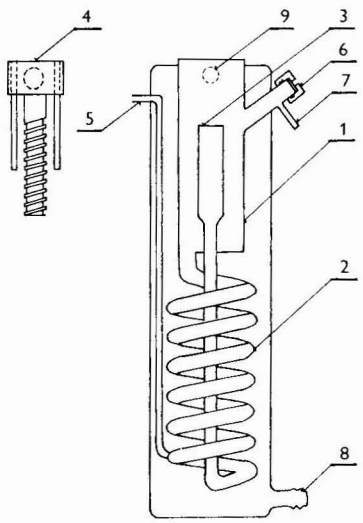


Fig. 2. Saturation vessel. 1 = Open flask; 2 = coil filled with glass beads; 3 = PTFE screw bush; 4 = PTFE screw; 5 = capillary of inert gas inlet; 6 = sampling device; 7 = vapour phase outlet; 8 = thermostated water inlet; 9 = thermostated water outlet.

(I.D. 7 mm) was filled with glass beads 3–3.5 mm in diameter. The circulation of the solution through the coil (0.2 ml/sec) with a counter-flow of the inert gas was provided by a rotating PTFE screw applying a flow-rate of the inert gas such that equilibrium between the liquid and vapour phases could be established.

The inert gas was fed from a pressure vessel (1, Fig. 1) of volume 37.72 l, allowing precise control of the overpressure from 0.01 to 0.19 MPa.

With *n*-pentane the analysis was performed on a Chrom 4 gas chromatograph with a flame-ionization detector (FID). The column dimensions were 3.5 m × 3 mm I.D., the support was Chromaton N AW DMCS (0.125–0.160 mm) wetted with 5 wt. % of Apiezon L, the column temperature was 343.15°K, the carrier gas was nitrogen and the overpressure was 0.06 MPa. In the FID the flow-rates of hydrogen and air were 0.5 and 5 ml/sec, respectively. Analysis of carbon tetrachloride was carried out on a home-made gas chromatograph with a Carlo Erba HT 20 electron-capture

detector. The column dimensions were 2.4 m × 3 mm I.D., the packing was the same as above (0.1–0.125 mm), the column temperature was 338.15°K, and the carrier gas was nitrogen, free from oxygen and water, at a flow-rate of 0.33 ml/sec.

## RESULTS AND DISCUSSION

### *Testing the method*

The dependences of log $S_1(n_3^+)$ on $n_3^+$ or on log (1 — constant·$n_3^+$) were obtained experimentally (see Tables I and II, respectively), based on 20–30 analyses each. As the assumption of linearity of the dependences proved to be justified, the corresponding straight lines were constructed by applying the least-squares method, the slopes were determined and the error of the slopes was estimated[4]. For the pre-saturation method, the pre-saturator temperatures were calculated by using eqn. 18, which has been proved[3] not to introduce a significant error.

TABLE I

LOG $S_1$ USING VARIANT WITH PRE-SATURATION

System: *n*-pentane (1)–*n*-octane (2)–nitrogen (3). Conditions: $T^- = 293.15$°K; $P^- = 0.101325$ MPa; $n_2(0) = 0.79506$ mol; $T^+ = 293.43$°K; $P^+ = 0.103191$ MPa. Average flow-rate of nitrogen = 0.182 ml/sec. Slope: $\frac{\mathrm{d}\log S_1(n_3^+)}{\mathrm{d}n_3^+} = -0.295622 \pm 0.003529$.

| *Average value of* $n^+$ *(mol)* | *log* $S_1$ | | |
|---|---|---|---|
| | *1st charge* | *2nd charge* | *3rd charge* |
| 0 | 3.56632 | 3.55712 | — |
| 0.20408 | 3.48671 | 3.48825 | — |
| 0.27612 | 3.44932 | 3.45165 | 3.45309 |
| 0.34135 | 3.43783 | 3.43953 | — |
| 0.60994 | 3.37262 | 3.37087 | — |
| 0.77952 | 3.33304 | 3.32945 | 3.33126 |
| 0.85771 | 3.29403 | 3.29296 | — |
| 1.09646 | 3.23629 | 3.22575 | — |
| 1.16767 | 3.19117 | 3.20063 | 3.19526 |
| 1.31833 | 3.13956 | 3.15254 | 3.14605 |
| 1.38210 | 3.12613 | 3.13518 | 3.13387 |
| 1.74945 | 3.04805 | 3.02965 | — |
| 1.92961 | 2.98046 | 2.97823 | 2.98162 |

The dependence of the slope per unit amount of the solvent on the flow-rate of the inert gas (Table III) indicates the optimal nitrogen flow-rate to be $4 \cdot 10^{-6}$–$8 \cdot 10^{-6}$ mol/sec, or 0.1–0.2 ml/sec. The data necessary for the calculation of the $\gamma$ value from eqn. 17 or 19 were obtained as follows: the fugacities were calculated from the second virial coefficients, the $P_i^0$ and $v_k$ (molar critical volumes) values were determined according to Voňka *et al.*[5], the $B_{ii}$ (virial coefficient) values for the alkanes and $CCl_4$ were calculated according to McGlashan and Potter[6] and for nitrogen the value was estimated according to Brewer and Vaugh[7]; $B_{ij} \approx (B_{ii} + B_{jj})^{1/2}$; $H_{3,2}$ was assigned the approximate value 77.4 MPa (ref. 8). The errors in the determination of $f^0_{C_5}$, $f^0_{C_8}$, $f^0_{CCl_4}$ and $f^-$ were 0.85, 2.5, 0.2 and 0.21 %, respectively.

TABLE II

LOG $S_1$ USING VARIANT WITHOUT PRE-SATURATION

System: *n*-pentane (1)–*n*-octane (2)–nitrogen (3). Conditions: $T^- = 293.15°K$; $P^- = 0.101325$ MPa; $n_2(0) = 0.82268$ mol. Average flow-rate of nitrogen = 0.163 ml/sec.

$$\text{Slope: } \frac{\mathrm{d}\log S_1(n_3^+)}{\mathrm{d}\log\left[1 - \frac{1}{n_2(0)} \cdot \frac{f_2^0}{f^-} \cdot \frac{n_3^+}{A - \frac{f_2^0}{f^-} \cdot \frac{f_3^-}{H_{3,2}}}\right]} = 40.8361 \pm 0.3287.$$

| *Average value of* $\log\left[1 - \frac{1}{n_2(0)} \cdot \frac{f_2^0}{f^-} \cdot \frac{n_3^+}{A - \frac{f_2^0}{f^-} \cdot \frac{f_3^-}{H_{3,2}}}\right]$ | *log* $S_1$ | | |
|---|---|---|---|
| | *1st charge* | *2nd charge* | *3rd charge* |
| 0 | 3.35027 | 3.34982 | — |
| −0.000342 | 3.33143 | 3.33085 | 3.33062 |
| −0.000522 | 3.32163 | 3.32111 | 3.32330 |
| −0.000950 | 3.30442 | 3.30492 | 3.30527 |
| −0.004289 | 3.17162 | 3.17280 | 3.17223 |
| −0.004355 | 3.15770 | 3.16125 | 3.15995 |
| −0.005543 | 3.12368 | 3.12188 | — |
| −0.005587 | 3.11878 | 3.11633 | 3.11585 |
| −0.008285 | 3.01063 | 3.00556 | 3.00109 |

TABLE III

DEPENDENCE OF SLOPE ON INERT GAS FLOW-RATE

System: *n*-pentane (1)–*n*-octane (2)–nitrogen (3). Experiment temperature, 293.15°K; pre-saturator temperature, 293.43°K; experiment pressure, 0.101325 MPa.

| *Nitrogen flow-rate (ml/sec)* | $n_2(0) \cdot \frac{\mathrm{d}\log S_1}{\mathrm{d}n_3^+}$ *(mol)* |
|---|---|
| 0.097 | −0.23452 |
| 0.182 | −0.23504 |
| 0.200 | −0.23536 |
| 0.253 | −0.23365 |
| 0.290 | −0.21615 |
| 0.385 | −0.16900 |

The error of the determination of $n_2(0)$ is negligible and that of the determination of $A$ is about 0.05%. For an analysis of the errors, the dependences of the slope per unit amount of solvent on pressure and temperature were established experimentally by applying the optimal flow-rate (Tables IV and V, respectively). For absolute errors with measurements of temperature of $\Delta T \approx 0.02°K$ and pressure of $\Delta P \approx 70$ Pa, the inaccuracy in the temperature measurement leads to a relative error of 0.068% and the inaccuracy in the pressure measurement and stabilization results in a relative error of 0.073%.

Based on the results of measurements given in Tables I and II, the values of the activity coefficients were calculated for *n*-pentane and *n*-octane at 293.15°K and

TABLE IV

DEPENDENCE OF SLOPE ON THE SATURATOR PRESSURE

System: *n*-pentane (1)–*n*-octane (2)–nitrogen (3). Experiment temperature, 293.15°K; pre-saturator temperature, 293.43°K; flow-rate of $N_2$, 0.1–0.2 ml/sec.

| *Saturator pressure (kPa)* | $n_2(0) \cdot \frac{\mathrm{d}\log S_1}{\mathrm{d}n_3^+}$ *(mol)* |
|---|---|
| 100.0 | −0.23895 |
| 101.325 | −0.23504 |
| 103.3 | −0.23052 |

TABLE V

DEPENDENCE OF SLOPE ON THE EXPERIMENT TEMPERATURE

System: *n*-pentane (1)–*n*-octane (2)–nitrogen (3). Experiment pressure, 0.101325 MPa; flow-rate of $N_2$, 0.1–0.2 ml/sec.

| *Pre-saturator temperature (°K)* | *Saturator temperature (°K)* | $n_2(0) \cdot \frac{\mathrm{d}\log S_1}{\mathrm{d}n_3^+}$ *(mol)* |
|---|---|---|
| 288.47 | 288.15 | −0.19513 |
| 290.93 | 290.65 | −0.21458 |
| 293.43 | 293.15 | −0.23504 |
| 295.94 | 295.65 | −0.25754 |
| 298.45 | 298.15 | −0.28410 |

a mean molar fraction of $x_1 = 0.0001$. The error was derived from the statistical error of the slope and the errors of the various variables and effects. For the procedure without pre-saturation $\gamma_1 \approx 1.003 \pm 0.042$ and for the procedure with pre-saturation $\gamma_1 = 0.982 \pm 0.023$. These results are consistent with the assumed behaviour of the *n*-pentane–*n*-octane system; a value of 0.99 has been found[9] at 303.15°K. Thus it is possible to employ the procedure in question for the determination of $\gamma$ at very low concentrations.

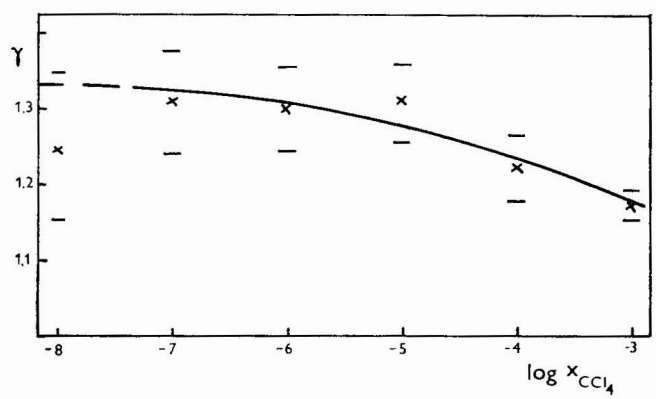


Fig. 3. Dependence of $\gamma_{CCl_4}$ on $CCl_4$ concentration.

*Concentration dependence of* $\gamma_{CCl_4}$

The above procedures were used to obtain the $\gamma_{CCl_4}$ values in *n*-octane at six different concentrations of carbon tetrachloride. When $x_{CCl_4} = 10^{-3}$ the method with pre-saturation was applied; the other systems were studied by using the variant without pre-saturation. The liquid phase was analysed and the dependence of log $S_1$-$(n_3^+)/S_1(0)$ on log (1 − constant·$n_3^+$) followed, where $S_1(0)$ is the peak area of the standard injected in succession (solution of carbon tetrachloride in *n*-octane of the same concentration as that of the solution measured at the beginning of the experiment). The errors of the individual measurements were determined as for the *n*-pentane–*n*-octane system and are plotted in Fig. 3. Extrapolation leads to an estimate of the limiting activity coefficient of carbon tetrachloride in *n*-octane at 293.15°K of $\gamma^{\infty}_{CCl_4} = 1.34$.

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# COMPONENT LOSS DURING EVAPORATION–RECONSTITUTION OF ORGANIC ENVIRONMENTAL SAMPLES FOR GAS CHROMATOGRAPHIC ANALYSIS

W. D. BOWERS and M. L. PARSONS*

*Arizona State University, Department of Chemistry, Tempe, AZ 85281 (U.S.A.)*

and

R. E. CLEMENT and F. W. KARASEK

*The Guelph-Waterloo Center For Graduate Work in Chemistry, Waterloo Campus, Department of Chemistry, Waterloo, Ontario N2L 3G1 (Canada)*

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## SUMMARY

Standard and sample solutions stored in borosilicate sample vials were allowed to evaporate to dryness at room temperature. The solutions were analyzed by gas chromatography–flame ionization detection before evaporation and after reconstitution to the original volume to determine component losses due to evaporation. The standard solutions were also stored in sample vials which had been treated with a surface deactivating agent, benzyltriphenylphosphonium chloride. The standard solution contained *n*-hydrocarbons, 1-alcohols, phthalates and polynuclear aromatic hydrocarbons. The sample solution was a benzene extract of municipal incinerator fly-ash which contained over 200 components including *n*-hydrocarbons, phthalates, polynuclear aromatic hydrocarbons and polychlorinated dibenzo-*p*-dioxins. At the 95% confidence level, the differences among mean losses observed with the 100 ng/$\mu$l standard mixture were within random variations between untreated and deactivated vials. The random variations between mean losses of the 10 ng/$\mu$l mixture were significantly higher with the deactivated vials at the 99% confidence level. Large losses were observed for early-eluting components of the standard solutions and the benzene extract of incinerator fly ash. Losses for polychlorinated benzo-*p*-dioxins and polynuclear aromatic hydrocarbons averaged *ca.* 10%.

## INTRODUCTION

Because of the high toxicity of certain substances, it is necessary to detect their presence in the environment at trace-to-ultratrace levels. These substances are usually present in mixtures containing a large number of components. The use of multi-step sample preparation and clean-up procedures in which the sample is taken to dryness and reconstituted before analysis is common[1,2]. The use of these procedures can result in the introduction of artifacts and loss of sample components. Karasek *et al.* have

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reported a rapid and simple procedure for the analysis of complex organic mixtures extracted from airborne particulate matter[3] and municipal incinerator fly ash[4] in which the sample clean-up steps are not necessary. Care is taken to prevent the sample extract from achieving dryness throughout the sample preparation procedure.

The method of reducing sample extracts to dryness and reconstituting to the final desired volume has been described[5–9]. It has been shown that significant losses result when pesticide residue extracts are evaporated to dryness before analysis[10,11]. Burke *et al.*[10] have investigated various concentration procedures used to bring samples to dryness and reported that losses were observed when the extract was concentrated to less than 500 $\mu$l, independently of the concentration procedure used. Chiba and Morley[11] reported that the use of petroleum ether as the extraction solvent resulted in greater losses upon condensation of the extract compared to benzene. In their study, detectable sample loss was observed even when a viscous retaining agent, such as ethylene glycol was used, when reducing the organic extract below 500 $\mu$l.

Although the Pyrex glassware generally used in trace analysis is considered inert, this surface has been observed to exhibit an undesirable activity towards polar compounds, owing to the presence of boron, potassium, and silanol groups in the glass matrix[12,13]. These active sites have been shown to adsorb polar compounds totally[13–15]. A common surface deactivation procedure is silylation. However, this only reacts with the silanol groups allowing the active metal sites to remain. Surface-active agents have been shown to be more effective in the deactivation of the entire glass surface towards polar compounds[12,15,16,18].

Studies reported to date which deal with sample component losses during concentration procedures have been primarily concerned with pesticide residues. Also, few data have been presented to show actual losses for other real samples, since most results have been obtained from standard solutions. As yet, no comprehensive study has been reported in which a large range of solvent– and sample–matrix systems have been investigated. This study was conceived to examine and compare component losses from mixtures containing a variety of compound types when evaporated to dryness and reconstituted. Component losses were investigated employing standard borosilicate sample vials, some of which were coated before use with benzyltriphenylphosphonium chloride (BTPPC) to achieve surface deactivation. Lowering the adsorptive properties of the glass surface might result in a greater recovery of sample components. Mixtures studied include a standard solution containing *n*-hydrocarbons, phthalates, polynuclear aromatic hydrocarbons (PAHs) and primary alcohols in cyclohexane as well as a benzene extraction of municipal incinerator fly ash. Of particular interest are the polychlorinated dibenzo-*p*-dioxins (PCDD), since some reported methods for their analysis require evaporation to dryness of sample extracts[1,2].

## EXPERIMENTAL

The concentrated standard solution used contained *n*-hydrocarbons, 1-alcohols, PAHs and phthalates in cyclohexane solvent. A dilute standard was prepared by a 1:10 dilution of the solution given in Table I. Straight-chain hydrocarbons and alcohols were from standard kits (Poly-Science, Niles, IL, U.S.A.), dioctyl phthalate was Baker "Practical Grade" (J. T. Baker, Phillipsburg, NJ, U.S.A.) and the other

phthalates were from Matheson, Coleman and Bell (Norwood, OH, U.S.A.) and PAHs were obtained from Aldrich (Milwaukee, WI, U.S.A.). Cyclohexane (Burdick & Jackson Labs., Muskegon, MI, U.S.A.) and benzene (Caledon Labs., Guelph, Canada) were "distilled-in-glass" grade. The BTPPC (Research Org./Inorg. Chem. Corp., Belleville, NJ, U.S.A.) surface-active agent was a 1% solution in methylene chloride ("distilled-in-glass" grade, Burdick & Jackson Labs.).

Storage containers used were Reacti-vials™ (Chromatographic Specialities, Brockville, Canada) equipped with screw-caps and PTFE liners. Before use, all glassware was cleaned by ultrasonic vibration in an aqueous solution of Alconox detergent (Alconox, New York, NY, U.S.A.), rinsed with copious amounts of tap water, rinsed thoroughly with deionized water, and heated in a laboratory oven for 1 h at 300°C. Glassware was allowed to cool to room temperature before use.

Vials to be deactivated were each coated five times with a 1% solution of BTPPC in methylene chloride. After each coating the vials were inverted and allowed to dry before the next application of BTPPC. All vials used in the study appeared to have even coatings.

*Standard solutions*

The experimental procedure followed for the standard mixtures is outlined in Fig. 1. A 1-ml volume of the concentrated (100 ng/$\mu$l) standard was placed into each of the four vials (two deactivated, two untreated). The dilute (10 ng/$\mu$l) standard was treated in the same manner. The original standard solutions were stored in a freezer at *ca.* −15°C. The solutions in the vials were allowed to evaporate to dryness by storing at room temperature in a fume hood with the screw-caps loosely fastened. The mixtures achieved dryness after *ca.* 20 h, and all vials were observed to have a yellow-brown residue which remained after evaporation. The standard solutions were then reconstituted by addition of 1 ml of cyclohexane. All vials were then agitated ultrasonically for *ca.* 1 min to promote homogeneity and redissolution of the organic residue. There appeared to be no residue after ultrasonic agitation.

*Incinerator fly ash extract*

The experimental procedure followed for the benzene extract of municipal incinerator fly ash is outlined in Fig. 2. The fly ash was supplied by the Ontario Ministry of the Environment and consisted of grab samples from municipal incinerators located in urban centers in southern Ontario. The fly ash, 116 g, was extracted with benzene using ultrasonic agitation. Initially, the fly ash was placed in a round-bottomed flask with 300 ml benzene and agitated for 30 min. After decanting the benzene through a medium-porosity glass-fritted filter, 100 ml of additional benzene was added and the 30 min extraction cycle was repeated. A total of four extraction cycles were used employing a total of 600 ml. After the last cycle the fly ash was transferred to the glass-fritted filter and rinsed with 50 ml of fresh benzene, and the sorbed fly ash extract was recovered by aspirator suction. The benzene extract was concentrated to a final volume of 800 $\mu$l by rotary evaporation and stored in a 1.0-ml vial equipped with screw-cap and teflon liner from which the 100-$\mu$l portions were taken for the sample evaporation study.

To each of two untreated vials was added 100 $\mu$l (Fig. 2). The remaining extract was stored in a freezer at *ca.* −15°C. The vials were allowed to evaporate to

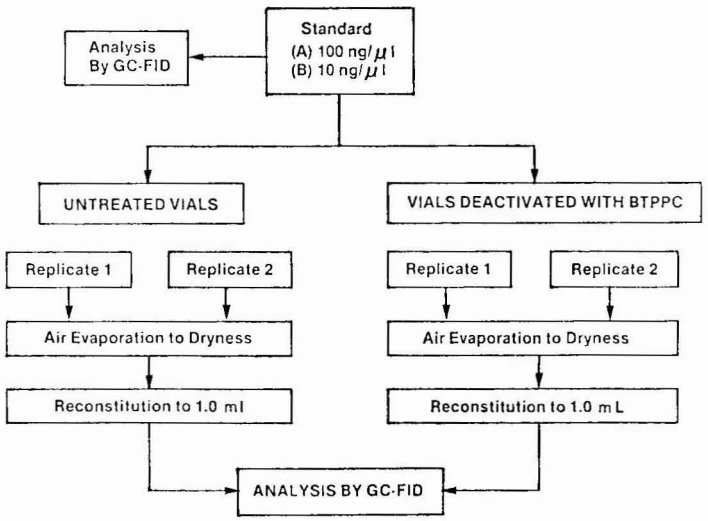


Fig. 1. Schematic of experimental procedure followed for the standard mixtures.

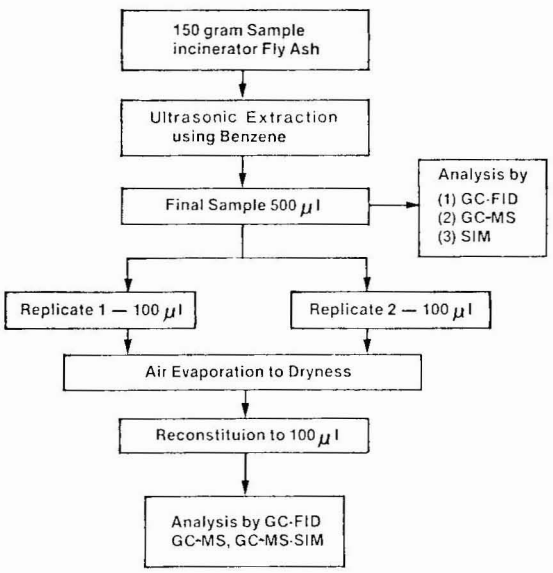


Fig. 2. Schematic of experimental procedure followed for the benzene fly ash extract.

dryness by storing at ambient temperatures in a fume hood with screw-caps loosely fastened. The extracts achieved dryness after *ca.* 40 h, and both vials were observed to have a yellow-brown residue. Each vial was then reconstituted by addition of 100 μl of benzene and then ultrasonically agitated for 1 min.

After reconstitution, all samples were analyzed by gas chromatography–flame ionization detection (GC–FID), using a Hewlett-Packard 5830A GC equipped with 1.8 m × 2 mm I.D. glass column packed with Aue packing[16]. Analysis conditions

were as follows: initial temperature, 90°C; program rate, 4°C/min; final temperature, 250°C, held for 15 min; injection port, 250°C; FID temperature, 275°C; helium carrier flow, 40 ml/min; injection volume, 3 $\mu$l. The initial temperature for the benzene sample condensates was 50°C. Original standards stored in the freezer were chromatographed under the same conditions for comparisons.

In addition to GC–FID analysis, original and reconstituted benzene condensates were analyzed by a Hewlett-Packard 5992 GC–MS–calculator system. GC conditions were as above with an initial temperature of 90°C. The GC–MS was operated in selected-ion-monitoring (SIM) mode, in which the quadrupole MS was selected tuned to each of six chosen ions during a single analysis. Compounds monitored were phthalates (ion 149.1), *n*-hydrocarbons (ion 85.1), biphenyl (ion 154.1), fluorene (ion 166.1), fluoranthene and pyrene (ion 202.2), anthracene (ion 178.1) and benzopyrene (ion 252.2). Various PCDD isomer series were also monitored, including the tetra (ion 321.9), penta (ion 355.9), hexa (ion 389.9) and hepta (ion 425.8) isomers, and octachlorodibenzo-*p*-dioxin (ion 459.7).

## RESULTS AND DISCUSSION

### *Evaporation–reconstitution of standard mixtures*

The loss of each component in the 100 ng/$\mu$l. standard mixture following evaporation–reconstitution is given in Table I. Comparison between the average integrated area of each component in the original mixture and the areas after the evaporation–reconstitution step was made to arrive at the amount of each component lost. The variance between the average mean losses of the deactivated and untreated vials were within random variations at the 95% confidence level. Variances were also compared between the first eight eluting components for both vials since they showed greater losses, and were within random variations at the 95% confidence level. This is also reflected by the average total loss from each type of vial which are within the injection and instrument variation of $\pm 3.1\%$.

The results of the evaporation–reconstitution procedure for the 10 ng/$\mu$l standard mixture are given in Table II. The first three eluting components were entirely lost by both vial types. The average mean losses between the deactivated and untreated vials were not within random variations at the 99% confidence level. This is an indication that the untreated vials were more reproducible in component loss following the evaporation step. The variations of the first eight eluting components were also greater for the deactivated vials at the 95% level, but the rest of the components were barely within the random variations at the 95% confidence level. The variability of the vials coating of BTPPC could be the major case of this observation.

Several deactivated vial values in Table II are reported as positive and correspond to a net gain in each component. These are due to random variations and are within the $\pm 1.8\%$ variation of injection and instrument fluctuations obtained from replicate injections of the original 10 ng/$\mu$l standard mixture. Comparisons of the average total loss observed for the deactivated and untreated vials indicates a significantly larger loss from the untreated vials. However, due to the large variance in the component recovery of the deactivated vials it is difficult to show that the use of deactivated vials will result in a lower loss of components.

TABLE I

COMPONENT LOSS AFTER EVAPORATION–RECONSTITUTION OF 100 ng/μl STANDARD SOLUTION

| *Component* | *Retention time (min)* | *Original solution (ng/μl)* | *Loss (ng/μl)* | | | |
|---|---|---|---|---|---|---|
| | | | *Deactivated No. 1* | *Deactivited No. 2* | *Untreated No. 1* | *Untreated No. 2* |
| Biphenyl | 3.6 | 126 | 66 | 63 | 61 | 50 |
| Fluorene | 7.7 | 102 | 28 | 28 | 30 | 21 |
| Dimethyl phthalate | 8.4 | 134 | 36 | 40 | 42 | 31 |
| Octadecane | 9.4 | 99 | 12 | 17 | 21 | 13 |
| Diethyl phthalate | 10.6 | 101 | 17 | 21 | 24 | 15 |
| Tetradecanol | 11.4 | 103 | 11 | 16 | 21 | 13 |
| Eicosane | 13.8 | 103 | 8 | 14 | 19 | 13 |
| Hexadecanol | 15.8 | 102 | 8 | 15 | 19 | 12 |
| Dibutyl phthalate | 18.0 | 102 | 8 | 14 | 19 | 12 |
| Fluoranthene | 20.0 | 111 | 11 | 17 | 21 | 16 |
| Tetracosane | 22.0 | 103 | 9 | 13 | 17 | 11 |
| Eicosanol | 23.7 | 104 | 13 | 15 | 18 | 11 |
| Hexacosane | 25.8 | 102 | 10 | 13 | 17 | 11 |
| Dioctyl phthalate | 28.9 | 101 | 6 | 13 | 17 | 10 |
| Triacontane | 32.8 | 100 | 6 | 11 | 16 | 11 |
| Benzo[*a*]pyrene | 35.9 | 100 | 11 | 16 | 15 | 11 |
| Total loss (ng/μl) | | | 260 | 326 | 377 | 261 |

TABLE II

COMPONENT LOSS AFTER EVAPORATION–RECONSTITUTION OF 10 ng/μl STANDARD SOLUTION

| *Component* | *Retention time (min)* | *Original solution (ng/μl)* | *Loss (ng/μl)* | | | |
|---|---|---|---|---|---|---|
| | | | *Deactivated No. 1* | *Deactivated No. 2* | *Untreated No. 1* | *Untreated No. 2* |
| Biphenyl | 3.6 | 12.6 | 12.6 | 12.6 | 12.6 | 12.6 |
| Fluorene | 7.7 | 10.2 | 10.2 | 10.2 | 10.2 | 10.2 |
| Dimethyl phthalate | 8.4 | 13.4 | 13.4 | 13.4 | 13.4 | 13.4 |
| Octadecane | 9.4 | 9.9 | 2.3 | 3.3 | 2.3 | 2.8 |
| Diethyl phthalate | 10.6 | 10.1 | 5.0 | 6.9 | 5.1 | 4.8 |
| Tetradecanol | 11.4 | 10.3 | 0.9 | 3.0 | 2.0 | 2.6 |
| Eicosane | 13.8 | 10.3 | +0.8* | 1.5 | 0.8 | 1.4 |
| Hexadecanol | 15.8 | 10.2 | +0.4 | 1.5 | 1.0 | 2.0 |
| Dibutyl phthalate | 18.0 | 10.2 | +1.2 | 0.8 | 0.4 | 1.1 |
| Fluoranthene | 20.1 | 11.1 | +0.3 | 2.1 | 1.4 | 2.1 |
| Tetracosane | 22.0 | 10.3 | +1.0 | 0.5 | 0.6 | 1.2 |
| Eicosanol | 23.7 | 10.4 | +1.3 | 0.5 | 0.7 | 1.5 |
| Hexacosane | 25.8 | 10.2 | +1.1 | 0.4 | 0.6 | 1.2 |
| Dioctyl phthalate | 28.9 | 10.1 | +1.0 | 0.5 | 0.5 | 1.0 |
| Triacontane | 32.5 | 10.0 | +0.9 | +0.1 | 1.3 | 1.8 |
| Benzo[*a*]pyrene | 36.0 | 10.0 | 0.1 | 2.3 | 1.3 | 1.6 |
| Total loss (ng/μl) | | | 36.5 | 59.4 | 54.2 | 61.3 |

* Positive value indicates a net gain in the component after evaporation–reconstruction.

*Evaporation–reconstitution of fly ash extract*

Fig. 3 is a comparison between the GC–FID results obtained for the original fly ash extract and one of the fly ash replicates after evaporation–reconstitution.

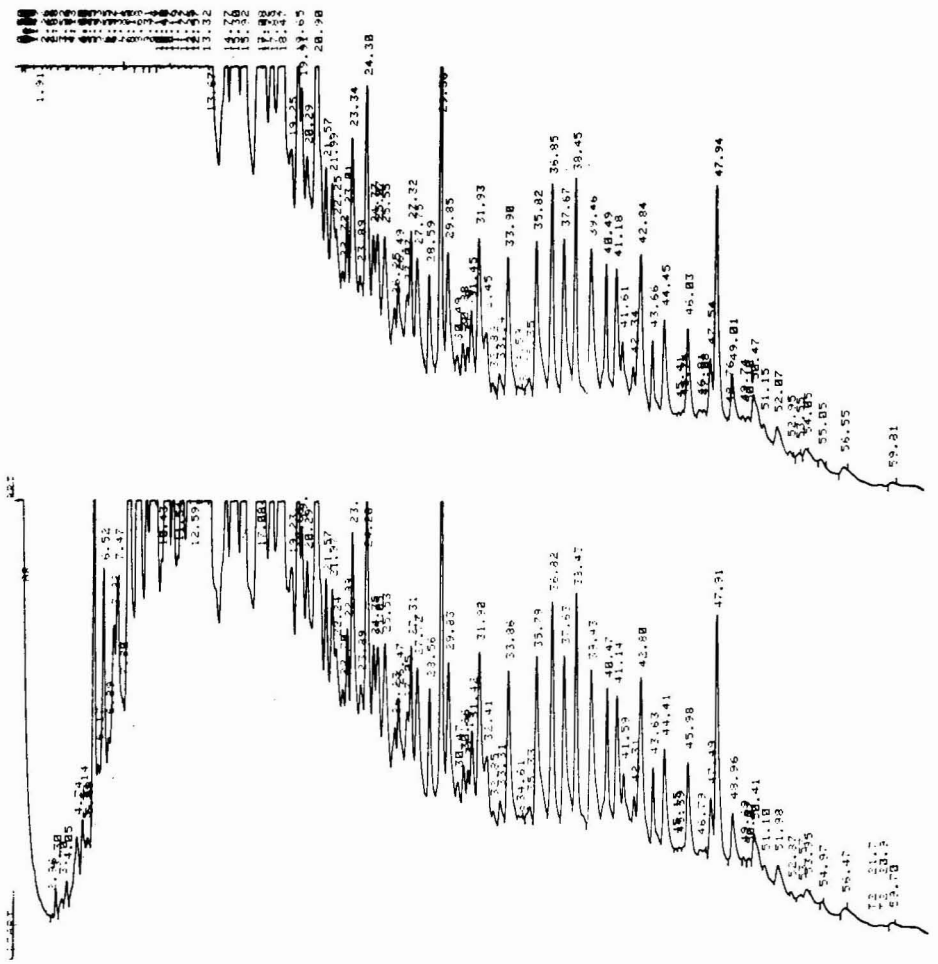

Fig. 3. Comparison of GC–FID results for 3-$\mu$l injections of: original fly ash extract (top), and the same extract after evaporation–reconstitution.

Although the two chromatograms are very similar for components which elute after a retention time of *ca.* 13 min, significant losses of earlier-eluting compounds can be observed in the reconstituted sample. Table III summarizes the GC–FID results for the original and evaporated–reconstituted replicates. Since peak areas can be related to amounts of substances through response factors, the data in Table III are a direct indication of the relative amounts of organic material detected.

Results of GC–MS analysis using SIM are given in Table IV. Reported recoveries for compounds in Table IV are based on comparisons between the average

TABLE III

GC–FID TOTAL AREAS* OF FLY ASH EXTRACT CONDENSATE BEFORE AND AFTER EVAPORATION–RECONSTITUTION

| | *Before evaporation to dryness* | | *After evaporation–reconstitution* | |
|---|---|---|---|---|
| | *Replicate I* | *Replicate II* | *Replicate I* | *Replicate II* |
| Early-eluting components (<retention index 1400) | 6516 | 8717 | 567 | 932 |
| Late eluting components (>retention index 2500) | 200 | 205 | 190 | 265 |
| Total area | 7579 | 10,000 | 3274 | 3797 |

* Areas are summations of total areas of all peaks detected and are normalized to largest total peak area = 10,000.

TABLE IV

RECOVERIES OF SELECTED COMPOUNDS AFTER EVAPORATION–RECONSTITUTION OF BENZENE EXTRACTION OF INCINERATOR FLY ASH

| *Compound* | *Recovery (%)* | |
|---|---|---|
| | *Replicate 1* | *Replicate 2* |
| *n*-Hydrocarbons ($C_{14}$–$C_{30}$) | 99 | 87 |
| Diethyl phthalate | 91 | 87 |
| Dibutyl phthalate | 92 | 91 |
| Dioctyl phthalate | 86 | 89 |
| Biphenyl | 67 | 62 |
| Pentachlorobenzene | 95 | 97 |
| Fluorene | 92 | 82 |
| Anthracene | 95 | 90 |
| Fluoranthene | 96 | 96 |
| Pyrene | 91 | 88 |
| Benzopyrene | 90 | 98 |
| Tetrachlorodioxins | 91 | 88 |
| Pentachlorodioxins | 89 | 86 |
| Hexachlorodioxins | 91 | 87 |
| Heptachlorodioxins | 97 | 80 |
| Octachlorodibenzo-*p*-dioxin | 90 | 80 |

integrated areas for two replicate injections of the original fly ash extract and the areas each of the evaporated–reconstituted samples. For the chlorinated dioxins, the total integrated areas for particular isomer series were compared. Identities of all of the compounds which are listed in Table IV were known by their mass spectra and correspondence of retention times of standards from previous work[4]. The average percentage deviation of areas from calculated means for the two non-evaporated samples was $\pm 2.3\%$, ranging from $\pm 0.02\%$ for biphenyl to $\pm 6.2\%$ for pentachlorobenzene. The corresponding average for the two evaporated–reconstituted replicates was $\pm 3.1\%$, which ranged from $\pm 0.2\%$ for fluoranthene to $\pm 10\%$ for the heptachlorodibenzo-*p*-dioxins. Percentage losses were less for the fly ash extract than for the standard solution for compounds common to both of these tests. Average

percentage losses for the standard solution were $15 \pm 2$, $16 \pm 3$ and $18 \pm 6$ for the *n*-hydrocarbons, phthalates and PAH compounds in the mixture, respectively. Corresponding average losses of $7 \pm 3$, $10 \pm 2$ and $8 \pm 5\%$ were observed for the corresponding *n*-hydrocarbons, phthalates and PAH which were detected in the fly ash extract. Biphenyl losses were not included in the above figures. They averaged 44% in the standard solution and 35% in the fly ash extract.

Most recoveries in Table IV are *ca.* 90%. The lowest recovery was achieved for biphenyl (65%), which is the lowest boiling compound of those in Table IV. These results indicate that bringing a sample to dryness may result in significant losses of extracted organics. Losses were observed even for high molecular weight components such as benzopyrene and the various chlorinated dioxin isomers. Since these samples were allowed to evaporate under very gentle conditions, lower recoveries may be expected when bringing a sample extract to dryness under conditions of reduced pressure and greater than ambient temperature. For this study, no sample transfer steps were involved other than the initial transfer of the stock solution to the sample vials. Further losses can be expected during regular sample analysis which may include several transfer steps and sample clean-up procedures.

ACKNOWLEDGEMENT

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# GAS CHROMATOGRAPHY OF MONOSACCHARIDES: FORMATION OF A SINGLE DERIVATIVE FOR EACH ALDOSE

HARTMUT FRANK*
*Institut für Toxikologie, Wilhelmstrasse 56, D-7400 Tübingen-1 (G.F.R.)*
and
HIGUINALDO JOSÉ CHAVES DAS NEVES* and ERNST BAYER
*Institut für Organische Chemie, Auf der Morgenstelle 18, D-7400 Tübingen-1 (G.F.R.)*
(Received November 4th, 1980)

## SUMMARY

A novel method for the derivatization of monosaccharides is presented which generates only one derivative for each aldose. It involves formation of aldoximes, their reduction with borane to the corresponding aminopolyols and subsequent conversion in to the N-ethoxycarbonyl-O-trimethylsilyl derivatives. Although there are four steps, only small amounts of side-products are found in the gas chromatograms. The derivatives are stable, at least for several days, and are well suited for determination of carbohydrates. For ketoses the same derivatization is applicable but results, as expected, in two diastereomers.

## INTRODUCTION

The unequivocal identification of monosaccharides is of general importance for structural elucidation of natural products and in biochemistry. Capillary gas chromatography is the method of choice for analysis of complex mixtures due to its high separation efficiency. In the case of sugars, however, complications arise from derivatization.

Carbohydrates themselves are not directly amenable to gas–liquid chromatography and require the preparation of appropriate volatile derivatives. Since the pioneering work of Bayer[1] and Sweeley[2] on the gas chromatographic (GC) properties of trimethylsilyl ethers of monosaccharides, oligosaccharides and sugar alcohols, many other derivatives have been proposed. Alditol acetates[3], trifluoroacetates[4], *n*-butyl boronates[5,6], aldonitriles[7], O-methyl glycoside trifluoroacetates[8], methoxime- and oxime-trimethylsilyl ethers[9,10], and anhydrohexose dithioacetals[11] have been used in the GC analysis of carbohydrates. The major problems with many monosaccharides and reducing oligosaccharides is the generation of isomeric com-

* Permanent address: Departamento de Quimica, Faculdade de Ciencias e Techologia, Universidade Nova de Lisboa, Quinta do Cabeço, 1899 Lisboa Codex, Portugal.

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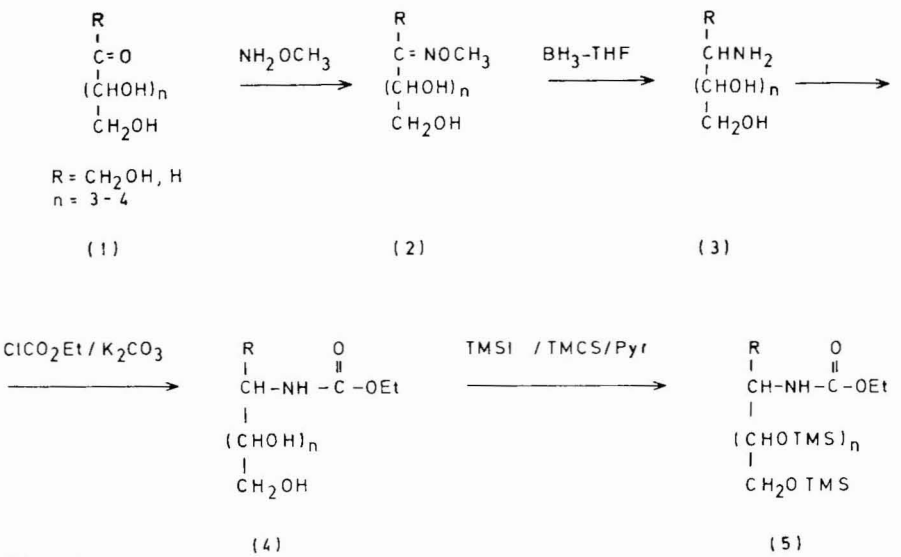
pounds during derivatization. This leads to multiple chromatographic peaks which interfere with the analysis of complex mixtures of carbohydrates. Peaks corresponding to different sugars may be superimposed, complicating their identification and quantitation[12]. Capillaries have been used in attempts to circumvent this difficulty[13–15]. The most successful approach to diminishing the number of peaks is the preparation of acyclic derivatives. Reduction to sugar alcohols, followed by acylation, has been used as a standard method for GC analysis of aldoses[3]. However, separation of some alditols is incomplete. Moreover, after reduction aldoses and ketoses afford identical alditols.




Scheme 1.

We now report a new method for the preparation of volatile acyclic derivatives of monosaccharides. Reduction of sugar methoximes yields aminodeoxyalditols (3), followed by ethoxycarbonylation (4) and trimethylsilylation to the N-ethoxycarbonyl-O-trimethylsilyl-aminopolyols (5) (Scheme 1). These derivatives are well separated in wall-coated open-tubular columns in a relatively short time. Aldoses give rise to only one peak, which allows their unequivocal identification and quantitation. For ketoses two well separated peaks of the corresponding diastereomers are obtained.

## EXPERIMENTAL

### *Materials*

Trimethylchlorosilane (TMCS) was obtained from Sigma (St. Louis, MO, U.S.A.), 1-trimethylsilylimidazole (TMSI) and ethyl chloroformate from E. Merck (Darmstadt, G.F.R.). Methoxyammonium chloride was purchased from Pierce (Rockford, IL, U.S.A.). Pyridine was dried over potassium hydroxide for 48 h,

refluxed over KOH and distilled. Samples of monosaccharides were purchased from Sigma.

*Apparatus*

Gas–liquid chromatography (GLC) was performed on a Dani instrument, Model 6800, equipped with splitter and flame ionization detector (FID). Two different capillaries were used: 25 m × 0.28 mm, coated with OV-101; and 25 m × 0.28 mm, coated with Chirasil-Val[16,17]. Injector temperature: 250°C. Detector temperature: 275°C. Oven temperature was programmed as shown in Figs. 2 and 3 at a rate of 1°/min. Mass spectrometry was performed on a Varian MAT 112 S instrument.

*Preparation of derivatives*

Aqueous standard solutions of the monosaccharides (each 1–2 mg/ml) with 2 mg/ml of D(−)-mannitol as internal standard were prepared. A 200-$\mu$l volume of each solution was transferred into a derivatization vial having a PTFE-lined rubber septum and screw-cap. The solvent was evaporated under a stream of nitrogen and the residue dried *in vacuo* over phosphorus pentoxide. A 100-$\mu$l volume of a solution of 250 mg of methoxyammonium chloride in 10 ml dry pyridine was added and the solution was heated to 80°C for 1 h. The solvent was evaporated under a stream of nitrogen or, if several samples were to be processed, in a vacuum centrifuge. A 100-$\mu$l volume of 1.0 *M* borane in tetrahydrofuran was added, the mixture agitated vigorously for 1 min and heated to 80°C for 2 h. After cooling to 0°C in an ice-bath, the excess of borane was destroyed by careful addition of methanol. The solvent was evaporated to dryness under nitrogen, 100 $\mu$l of 1 *M* HCl in methanol were added and the solution was heated to 80°C for 30 min. After cooling, the solvent was evaporated under a stream of nitrogen and the operation was repeated. The residue was dissolved in 50 $\mu$l of a saturated aqueous solution of $K_2CO_3$ and 25 $\mu$l of ethyl chloroformate were added. The mixture was vigorously agitated for 1 min and left at room temperature for 1 h. The liquid was then evaporated under nitrogen and the residue dried overnight *in vacuo* over phosphorus pentoxide. The dry residue was taken up in 40 $\mu$l of dry pyridine, 10 $\mu$l of trimethylchlorosilane (TMCS) and 10 $\mu$l of trimethylsilylimidazole (TMSI), agitated vigorously for 2 min and heated to 50°C for 30 min. After centrifugation, the supernatant was used for GC.

*Calculation*

Peak areas were calculated by means of a Spectra-Physics SP 4100 electronic integrator. The response factor of each sugar was calculated relative to D(−)-mannitol.

## RESULTS AND DISCUSSION

Gas chromatography of sugars can greatly be simplified by the use of acyclic derivatives. However, for methoxime derivatives, two peaks are observed for each sugar[9], *i.e.*, the *syn*- and *anti*-forms. Oxime ethers can readily be reduced by borane in tetrahydrofuran to give the corresponding amines in high yields[18]. Borane offers a distinct advantage over lithium aluminium hydride: aluminium hydroxide is a strong adsorbent, and the yields of reduction are generally low[19]. The reaction conditions of

the borane reduction have not been optimized with respect to time and temperature. Considering the reactivity of borane, complete reduction may be achieved at lower temperatures and in shorter times.

The obtained aminopolyols can further be derivatized by different methods. Silylation was found to be difficult or gave two or more peaks with bis(trimethylsilyl)-trifluoroacetamide (BSTFA) as silylating agent. This can be attributed to the fact that BSTFA can introduce either one or two silyl groups in primary amines[20]. On the other hand, the trifluoroacetyl derivatives were found to be unstable, and the more stable pentafluoropropionyl derivatives were not well separated.

Amino sugars can readily be converted with ethyl chloroformate into the corresponding N-ethoxycarbonyl derivatives[21]. These are stable and, after silylation, exhibit excellent GC properties. For silylation a mixture of pyridine–trimethylchlorosilane–trimethylsilylimidazole (4:1:1) gave the best results.

Identity of the products obtained was established by gas chromatography-mass spectrometry (GC–MS) of the derivatives obtained by reduction with trideuterioborane. The fragmentation patterns observed are analogous to those of the trimethylsilyl derivatives of the sugar oximes and methoximes. The mass spectrum of the glucose derivative (Fig. 1) shows no molecular ion, but a relatively intense $[M - 15]^+$ (*m/e* 600), corresponding to incorporation of two deuterium atoms, several chain cleavage fragments (*m/e* 103, 205, 307, 409, 410) and some cascades of ions from

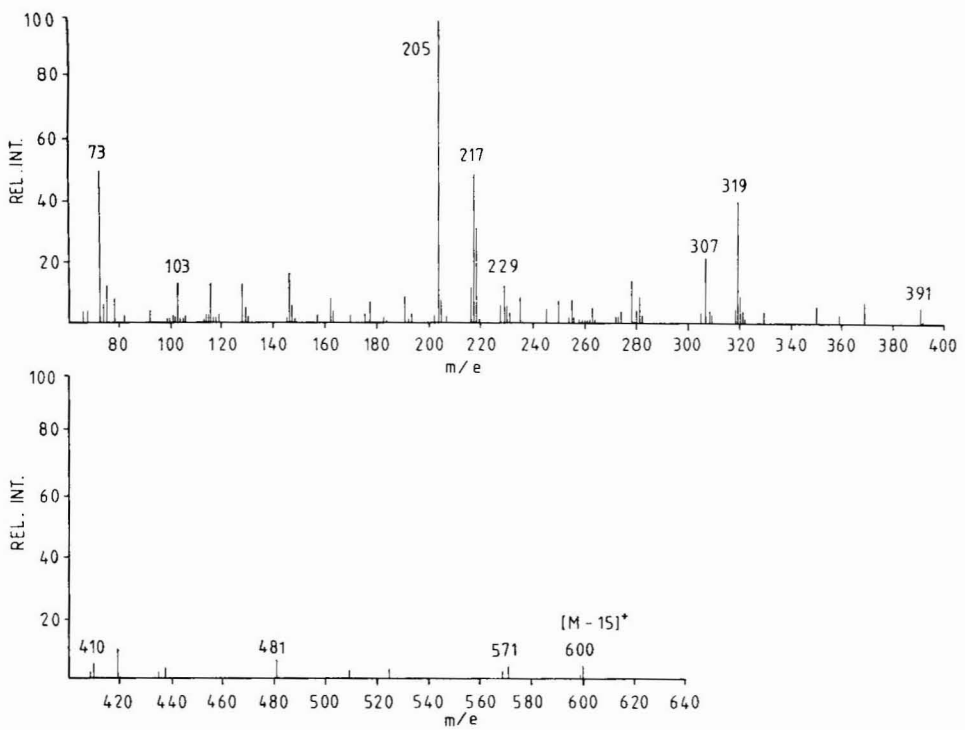



Fig. 1. Electron-impact (EI) mass spectrum of the N-ethoxycarbonyl-O-trimethylsilyl-aminodeoxyalditol of glucose. Conditions: electron energy 70 EV; anode current 0.7 mA; GC-interface temperature 250°C; ion source temperature 270°C.

sequential elimination of trimethylsilanol (571–481–391, 600–509–419–329, 410–319–229, 307–217). Other diagnostic ions are at *m/e* 571 ($[M - 44]^{+\cdot}$) and *m/e* 73 (trimethylsilyl).

Retention times for the derivatives of arabinose, ribose, rhamnose, fucose, fructose, galactose, mannose and glucose were determined in capillaries coated with Chirasil-Val (Fig. 2) and OV-101 (Fig. 3). As can be seen from the chromatograms, only one derivative is formed for each carbohydrate. The retention times on Chirasil-Val are relatively short with good separation of all sugars, except for galactose and mannose. A good separation of all sugars is obtained with OV-101, but with significantly higher retention times. However, if analysis is carried out isothermally at 180°C separation is achieved in 20 min. Interestingly, an inversion of the elution order of rhamnose and fucose is observed on changing from Chirasil-Val to OV-101 (peaks 4 and 5).

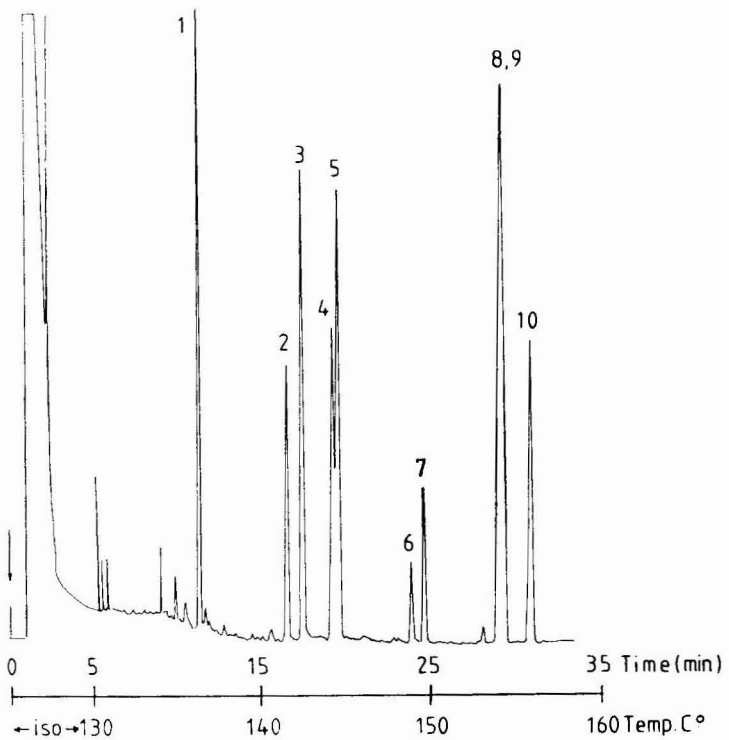



Fig. 2. Gas chromatogram of N-ethoxycarbonyl-O-trimethylsilyl-aminodeoxyalditols on Chirasil-Val (25 m × 0.28 mm). Carrier gas hydrogen: 0.4 kg/cm². Splitting ratio 1:25. Injector temperature: 250°C. FID temperature: 275°C. Peaks: 1 = TMS-mannitol as standard; 2 = arabinose; 3 = ribose; 4 = rhamnose; 5 = fucose; 6 = fructose 1; 7 = fructose 2; 8 = galactose; 9 = mannose; 10 = glucose.

In contrast to the aldoses, the ketosugar fructose, as expected, shows two well separated peaks, corresponding to the two possible diastereomers formed on reduction of the fructose methoxime. The reaction obviously proceeds with some stereoselectivity as the two peaks of fructose have an area ratio of *ca.* 1:2. Although

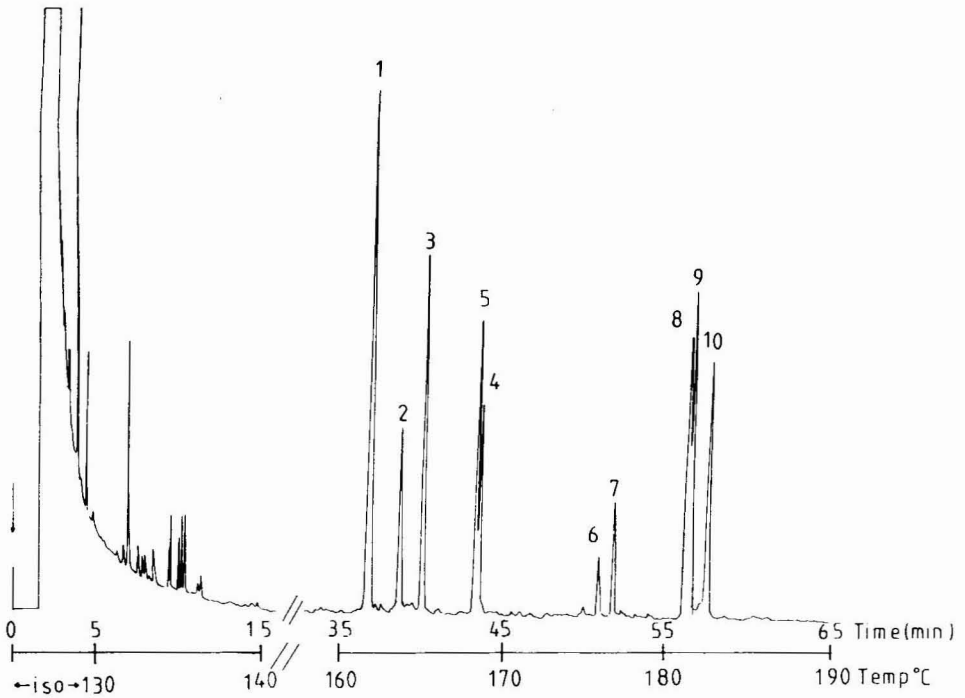



Fig. 3. Chromatogram of the N-ethoxycarbonyl-O-trimethylsilyl-aminodeoxyalditols on OV-101 (25 m × 0.28 mm). Conditions and numbering of peaks as in Fig. 2.

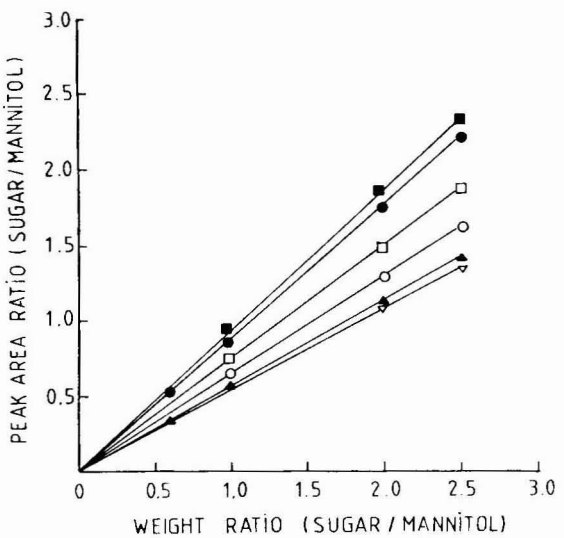



Fig. 4. Gas chromatographic response of N-ethoxycarbonyl-O-trimethylsilyl-aminodeoxyalditols relative to TMS-mannitol as internal standard: ■, galactose; ●, ribose, rhamnose; □, fucose, mannose; ○, glucose; ▲, arabinose; ▽, fructose (sum of both peak areas).

formation of two derivatives constitutes a complication in the GC analysis of ketoses, the good separation of both peaks still allows quantitation. In any case, the proposed derivatization considerably simplifies the GC pattern[22].

Relative response factors were determined for each sugar, from mixtures containing various amounts of sugar and a fixed amount of mannitol as internal standard. Good linearity was observed (Fig. 4), but the detector response for the sugar derivatives is somewhat lower than for TMS-mannitol. A similar observation has been made previously[21].

## CONCLUSIONS

The GC analysis of carbohydrates is greatly facilitated by a derivatization sequence which affords only one derivative for aldoses. In the case of ketoses two diastereomers are obtained. The derivatives are very stable and exhibit excellent GC properties. Most other derivatization procedures yield either more than one peak and/or easily decomposing derivatives. For instance, TMS-tagatose gives rise to seven or eight components[22]. The derivatization sequence involves four consecutive reactions, but offers the great advantage of unambiguous identification and simplified quantitation of sugars.

The identity of the compounds has been established by GC–MS. Formation of side- or decomposition products is negligible; consequently the sample amount required is low: derivatization was performed with about 1 $\mu$mol; GC was carried out with 0.1–1 nmol. We consider that this method is a significant contribution towards a more sensitive and accurate analysis of carbohydrates in natural products and biochemical samples.

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# QUANTITATIVE GAS CHROMATOGRAPHIC ANALYSIS OF SUCROSE IN THE PRESENCE OF SUGAR OXIMES USING A BUFFERED OXIMATION REAGENT AND GLASS CAPILLARY COLUMNS

K. J. SCHÄFFLER* and P. G. MOREL DU BOIL
*Huletts Sugar Limited, Research & Development, P.O. Mount Edgecombe, 4300 Natal (South Africa)*
(Received September 2nd, 1980)

## SUMMARY

Aqueous sugar solutions containing fructose, glucose and sucrose can be derivatised rapidly with a novel oximating reagent, incorporating an organic buffer. The buffered reagent produces rapid and reproducible oximation of the monosaccharides without hydrolysing sucrose or affecting its silylation.

Reasons for the use of a SP-2250 glass capillary for the separation of sugars in cane molasses are also given.

## INTRODUCTION

Sucrose, fructose and glucose are the main carbohydrates present in sugar cane juice and subsequent factory processing streams. Their estimation is important in assessing both sugar cane quality and factory performance[1]. The approximate ranges of the three sugars during processing are listed in Table I.

TABLE I

RANGE OF SUGAR LEVELS IN CANE SUGAR FACTORY STREAMS

Concentrations are expressed as % sugar in sample.

| *Stream* | *Fructose (F)* | *Glucose (G)* | *F/G* | *Sucrose* |
|---|---|---|---|---|
| Mixed juice | 0.2–0.6 | 0.2–0.6 | 1.0 | 9–13 |
| Syrup | 1–2 | 1–2 | 0.9–1.1 | 50–60 |
| Final molasses | 6–11 | 3–9 | 1.1–2.4 | 26–33 |

Gas–liquid chromatography (GLC) provides a means of measuring these three sugars more specifically than traditional titration and polarisation techniques which are readily influenced by impurities[2,3].

The volatilisation of sugars prior to GLC is normally achieved by converting the sugars into their trimethylsilyl (TMS) derivatives. The TMS ethers of monosaccharides possess the following disadvantages:

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(1) the proportions of each anomer will depend on solvent composition, temperature and the length of time the sugar has been dissolved;

(2) the overlap between fructose and glucose leads to inaccurate results;

(3) the overlap of the two major monosaccharides with other minor constituents in cane molasses will also give inaccurate results;

(4) the signal-to-noise ratio for a monosaccharide producing multiple peaks is obviously lower than a sugar producing a single peak. This is extremely important when the sugar is present in low concentration (see Table I).

Various chemical procedures have been proposed to inhibit anomerisation. Sweeley *et al.*[4] suggested oximation prior to silylation. Brobst[5] developed an *in situ* oximation–silylation procedure for sugars in aqueous solution. The reagents for this procedure are available commercially from a single supplier[6].

Aqueous sugar solutions covering a wide concentration range (0.5–35%) were derivatised in this laboratory, and although excellent qualitative results were obtained we noticed that sucrose (a non-reducing disaccharide and the sugar of prime importance to the sugar technologist) was hydrolysed during the oximation of fructose and glucose. This was due to the acidity of the oximation reagent.

This paper describes an oximation reagent incorporating an organic buffer. The buffered reagent produced rapid and reproducible oximation of monosaccharides without hydrolysing sucrose. Xylose and trehalose were added as internal standards for the monosaccharides and sucrose respectively. Reasons for using an SP-2250 glass capillary column are also given.

## EXPERIMENTAL

### *Materials*

Fructose (low in glucose), glucose (AnalaR), sucrose (Aristar), xylose (Biochemical) and trehalose dihydrate (Biochemical) were obtained from BDH (Poole, Great Britain). All reference sugars were dried *in vacuo* over phosphorus pentoxide and stored in a desiccator. The following reagents were commercially available: pyridine (for analysis; E. Merck, Darmstadt, G.F.R.); hydroxylamine hydrochloride (M & B reagent; May & Baker, Dagenham, Great Britain); hexamethyldisilazane (HMDS) (Ohio Valley, Manetta, OH, U.S.A.), stored under nitrogen and refrigerated; trifluoroacetic acid (TFA) (Pierce, Rockford, IL, U.S.A.), refrigerated; dimethylaminoethanol (BDH laboratory reagent).

### *Preparation of derivatives*

*Hydrolysis of sucrose during oximation.* A calibration solution was prepared by dissolving sucrose (600 mg) and trehalose (660 mg) in 2.4 ml of distilled water (solution A). Aliquots were silylated directly or oximated prior to silylation.

The aliquot (5 $\mu$l) in a screw cap vial (3 ml) fitted with a PTFE-lined silicon disc was silylated by adding pyridine (0.5 ml), HMDS (0.45 ml) and TFA (0.05 ml) in rapid succession. The vial was hand shaken, capped and placed in an ultrasonic bath at 80°C for 10 min. The sample was degassed prior to injection.

An aliquot (5 $\mu$l) in a 3-ml vial was treated with 0.5 ml of oximation reagent (2.5 g of hydroxylamine hydrochloride in 100 ml of pyridine). The sample was placed in an ultrasonic bath at 80°C for 30 min. After cooling for 10 min, HMDS (0.45 ml) and TFA (0.05 ml) were added. The silylation was carried out at 80°C for 10 min.

*pH of oximation reagent.* Aliquots (5 $\mu$l) of solution A were treated with various amounts of dimethylaminoethanol (0, 9, 18 and 27 $\mu$l), followed by the oximating reagent in pyridine (0.5 ml) (2.5%, w/v). Oximation and silylation were carried out as above.

*Effectiveness of buffered oximation reagent.* A calibration standard containing the following sugars was prepared: fructose, 150 mg; glucose, 150 mg; xylose, 150 mg and water, 2.4 ml. Aliquots (in triplicate) were treated with various amounts of dimethylaminoethanol (0, 18 and 27 $\mu$l), followed by oximation and silylation as above.

*Effect of new buffered oximation reagent on sucrose hydrolysis.* Three calibration standards bracketing the sucrose concentration range for cane molasses were prepared:

| | $S_1$ | $S_2$ | $S_3$ |
|---|---|---|---|
| Trehalose (mg) | 660 | 660 | 660 |
| Sucrose (mg) | 500 | 600 | 700 |
| Water (ml) | 2.4 | 2.4 | 2.4 |

Aliquots were silylated in triplicate above. The buffered oximation reagent was prepared by adding 2.5 g of hydroxylamine hydrochloride to pyridine (100 ml). Dimethylaminoethanol (270 $\mu$l) was added to 5 ml of this hydroxylamine solution just before it was needed. The solution was mixed thoroughly (solution B). Aliquots for oximation were prepared in triplicate. Oximation with solution B was identical to the procedure described above.

*Gas chromatography.* A Hewlett-Packard 5840 gas chromatograph equipped with an autosampler was employed. Experimental details are listed in Table II.

TABLE II

EXPERIMENTAL DETAILS FOR GLC SEPARATION OF OX–TMS SUGAR DERIVATIVES

| | |
|---|---|
| Column | 15 m × 0.25 mm I.D. glass capillary, coated with SP-2250 (obtained from SGE, North Melbourne, Australia) |
| Inlet pressure | 25 kPa |
| Pre-column flow-rate | 10.6 cm$^3$/min |
| Column flow-rate | 0.4 cm$^3$/min (nitrogen) |
| Split ratio | 25:1 |
| Injection volume | 4 $\mu$l (0.15 $\mu$l onto column) |
| Injector/flame ionization detector temperature | 250/250°C |
| Oven program | 150°C for 2 min, 150–240°C at 8°C/min |
| Make-up gas | 60 cm$^3$/min (nitrogen) |
| Hydrogen | 40 cm$^3$/min |
| Air | 330 cm$^3$/min |

## RESULTS AND DISCUSSION

### *Sucrose hydrolysis*

Use of Brobst's procedure to oximate an aqueous sucrose–trehalose mixture prior to silylation produced a lower sucrose response factor (1.075) than that obtained

for direct silylation (1.097). Fig. 1A clearly indicates significant sucrose hydrolysis, as fructose and glucose can be detected easily. The pH of the hydroxylamine reagent was found to be 5.4, Stadler's table[15] indicates about 0.15% sucrose inversion per hour at 80°C at this pH.

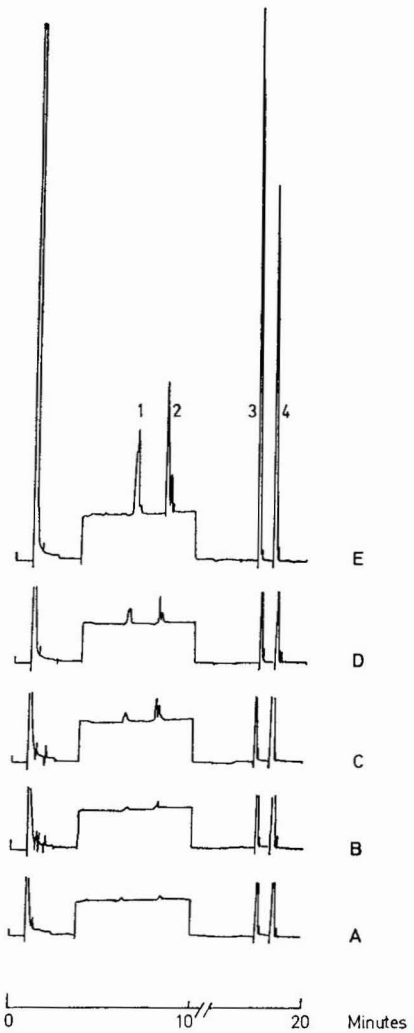


Fig. 1. Sucrose hydrolysis during oximation: E = unbuffered oximation reagent, pH = 5.4; D = buffered oximation reagent, pH = 6.5; C = buffered oximation reagent, pH = 7.1; B = buffered oximation reagent, pH = 7.4; A = direct silylation (no oximation). Peaks: 1 = OX–TMS-fructose; 2 = OX–TMS-glucose; 3 = TMS–sucrose; 4 = TMS-trehalose. Attenuation: for monosaccharides, 2 ↑ 3; for disaccharides, 2 ↑ 7.

To ascertain whether 80°C was necessary for oximation, lower temperatures were investigated. At 50°C and a reaction time of 30 min, oximation was incomplete. At this temperature sucrose hydrolysis was still apparent. No reference to such

hydrolysis has been noted by us. Adam and Jennings[7], using methyl oximes, noticed fructose and glucose peaks in all their chromatograms; they attributed this to sucrose hydrolysis during their drying step (using phosphorus pentoxide).

*Buffer*

Although oximation is acid-catalysed and hydroxylamine is unstable in basic solution, Fritz *et al.*[8] developed a quantitative titrimetric procedure for carbonyl compounds employing semi-neutralised hydroxylamine hydrochloride solutions. The choice of buffer was limited by the anhydrous conditions and the fact that the hydrochloride of the base used should be soluble in the solvent. These workers found either 2-dimethylaminoethanol or 2-diethylaminoethanol to be suitable.

*Effect of pH of the oximation reagent on sucrose hydrolysis*

Dimethylaminoethanol was used to raise the pH of the oximation (OX) reagent without causing any undesirable precipitation reactions. The effect of neutralising the OX reagent before oximation can be seen in Fig. 1 and Table III.

TABLE III

EFFECT OF pH OF OXIMATION REAGENT ON SUCROSE RESPONSE FACTOR

Base = Dimethylaminoethanol. B:A = Equivalents base relative to equivalents HCl. Response factors, *K*, are the means of three sample preparations. R.S.D. = Relative standard deviation.

| *Base (μl)* | *B:A* | *pH* | *Expected hydrolysis (%/h)* | *K* | *R.S.D.* |
|---|---|---|---|---|---|
| 0 | 0 :1 | 5.4 | ≈0.15 | 1.077 | 0.9 |
| 9 | 0.5:1 | 6.5 | 0.01 | 1.093 | 0.7 |
| 18 | 1 :1 | 7.1 | 0.003 | 1.095 | 0.1 |
| 27 | 1.5:1 | 7.4 | <0.001 | 1.097 | 0.3 |
| — | Direct silylation | — | — | 1.097 | 0.3 |

The response factor for sucrose using an OX reagent at a pH of 7.4 was virtually identical to that obtained for direct silylation. Fig. 1 also indicates that with the buffered reagent sucrose hydrolysis was not significant. Any detectable quantities of fructose and glucose are probably minute impurities in Aristar sucrose. No significant hydrolysis of other disaccharides such as trehalose, maltose or cellobiose was observed when using Brobst's procedure.

*Effect of pH on monosaccharide oximation*

Buffering the oximation reagent eliminated sucrose hydrolysis. The effect of increasing the pH of the oximation reagent on the actual oximation of fructose and glucose is noted in Table IV. It is obvious that effective oximation of fructose and glucose can be obtained even in slightly alkaline solution, as excellent agreement over the range pH 5.4–7.4 was obtained.

TABLE IV

EFFECT OF pH OF OXIMATION REAGENT ON FRUCTOSE AND GLUCOSE RESPONSE FACTORS

Number of samples in each case: 12. Oximation time = 30 min. Internal standard: xylose. These response factors are the means of three sample preparations.

| *pH* | $K_F$ | *R.S.D.* | $K_G$ | *R.S.D.* |
|---|---|---|---|---|
| 5.4 | 1.072 | 0.2 | 1.058 | 0.1 |
| 6.5 | 1.076 | 0.3 | 1.058 | 0.1 |
| 7.1 | 1.075 | 0 | 1.058 | 0.1 |
| 7.4 | 1.076 | 0.3 | 1.058 | 0.2 |
| Mean | 1.075 | 0.3 | 1.058 | 0.1 |

*Decreasing oximation reaction time*

We have shown that excellent quantitation of fructose and glucose can be obtained by using a slightly alkaline oximation reaction prior to silylation. For all these studies an OX reaction time of 30 min at 80°C was adopted. To reduce sucrose hydrolysis even further, an OX reaction time of 10 min at 80°C was investigated. It can be seen from Table V that statistically there was no difference between the two reaction times. This reduction in sample preparation time is obviously advantageous for routine high throughput analysis.

TABLE V

EFFECT OF OX REACTION TIME ON MONOSACCHARIDE RESPONSE FACTORS

Numbers of samples: 12. Mean difference between response factors at different OX reaction times, $\bar{D} = 0.001$. $t_{exp}$ = Calculated Student's $t$ value for paired observations. Critical $t$ value, $t_{1-\alpha}$, ($\alpha = 0.05$) = 2.776.

| | $K_F$ | $K_G$ |
|---|---|---|
| Time | | |
| 30 min | 1.075 | 1.058 |
| 10 min | 1.076 | 1.058 |
| $t_{exp}$ | −0.06 | −0.11 |

*Comparison of sucrose response factors: direct silylation vs. OX/silylation*

Accurate sucrose analysis should be independent of monosaccharide oxime formation. The effect of the new buffered OX reagent was investigated by preparing three aqueous sucrose–trehalose standards, bracketting our normal cane molasses sample range (see Experimental). The comparison is presented in Table VI. There was

TABLE VI

SUCROSE CALIBRATION STANDARDS FOR CANE MOLASSES

Number of samples: 12. $t_{exp} = -1.91$; $t_{1-\alpha}$ ($\alpha = 0.05$) = 2.31.

| | $K_S$ |
|---|---|
| Direct silylation* | 1.106 |
| OX–silylation** | 1.109 |

* Silylation conditions: 10 min at 80°C.

** OX time: 10 min at 80°C followed by silylation for 10 min at 80°C.

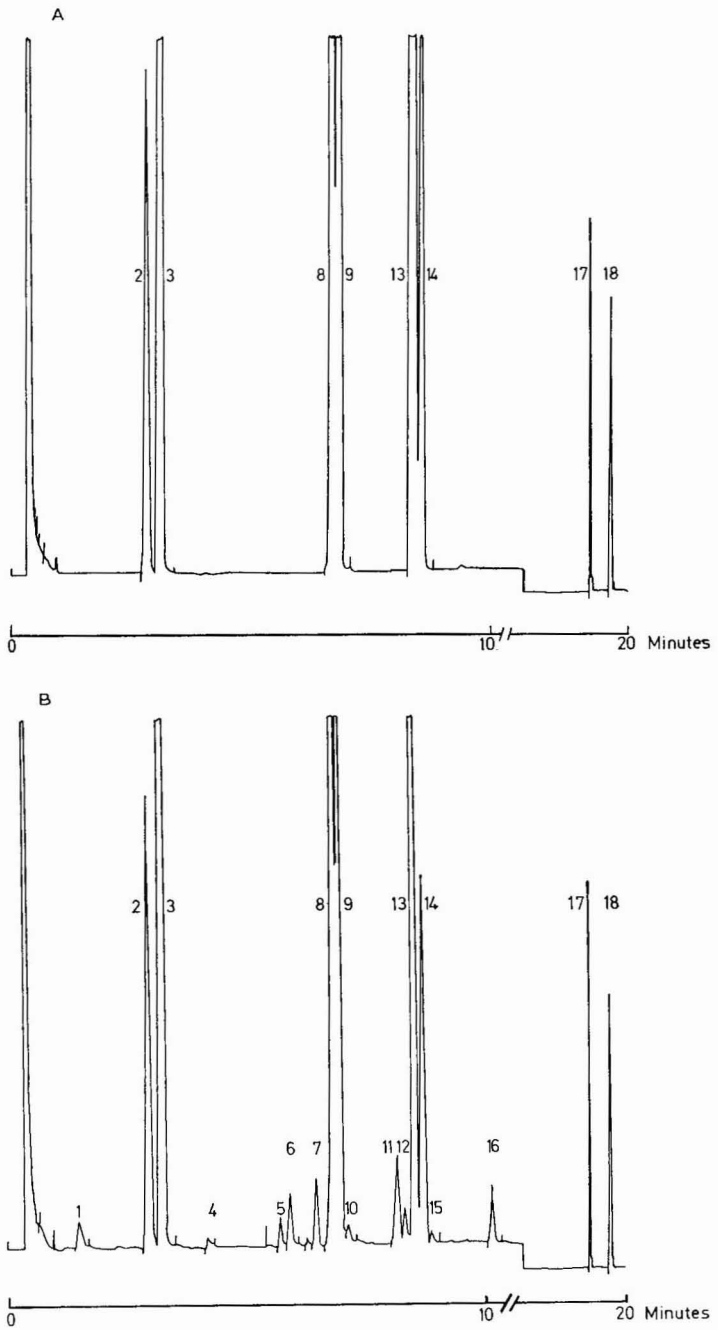


Fig. 2. Separation on SP-2250 (OV-17) wall-coated glass capillary columns: A = Calibration standard; B = cane molasses sample. OX–TMS derivatives: 2,3 = xylose; 8,9 = fructose; 13,14 = glucose; 17 = sucrose; 18 = trehalose. Other peaks: 6 = mannitol; 11 = mannose; 16 = inositol; 1, 4, 5, 7, 10, 12, 15 = unknowns. Chart speed: for monosaccharides, 2.5 cm/min; for disaccharides, 0.25 cm/min. Attenuation for mono- and disaccharides was 2 ↑ 5 and 2 ↑ 7, respectively.

no statistical difference and in our opinion the new buffered reagent effectively produces monosaccharide oximes without hydrolysing sucrose.

*Epimerisation of monosaccharides at pH 7.5*

In dilute alkaline solutions the monosaccharides can undergo profound changes[9]. Glucose can produce its epimers mannose and fructose. To investigate the effect of the higher pH (7.4) of the oximation reagent on monosaccharide epimerisation, dilute aqueous solutions containing either fructose or glucose were subjected to the OX–TMS procedure at pH 7.4. No evidence of epimerisation was observed.

*Use of glass capillary columns*

Packed columns were used initially during the development of the GC procedure for sugars[3,10]. We recently utilised stainless-steel capillaries coated with OV-17 for an extensive study of carbohydrate changes during sugar boiling[11]. However, glass capillaries offer certain advantages due to better peak separation and sharper peaks: greater column efficiencies; better column deactivation; often better resolution in less time; more information and more accurate results.

We therefore switched to glass capillaries. SP-2250 (OV-17) wall-coated columns produced an excellent separation of OX–TMS-xylose (internal standard), fructose, glucose, TMS-sucrose and trehalose (internal standard), Fig. 2A. Carrier gas flow was optimised (average flow velocity, 16 cm/sec), thus all OX–TMS sugars produced acyclic *syn* and *anti* isomers. Despite the separation of each monosaccharide into its respective doublets, each sugar was well separated.

An aqueous molasses sample was subjected to the OX–TMS procedure, Fig. 2B. Besides fructose and glucose, approximately ten minor constituents could also be observed in the molasses sample. We have been monitoring sugar products for over 3 years and the pattern depicted in Fig. 2B is completely characteristic of South African cane molasses. Some of these peaks, mannitol, mannose and inositol, have been identified. The use of columns with lower efficiency and selectivity can result in the overestimation of both fructose and glucose by about 6–11% in South African cane molasses.

A further advantage of the use of capillary columns is their ability to separate the geometric isomers of each monosaccharide OX–TMS derivative. The peak area ratio under controlled conditions is characteristic for the isomers of each sugar. A change in this area ratio can often indicate an impurity co-eluting with one or the other isomer. This is of practical importance for low concentrations of fructose and glucose.

Several authors have indicated that OX/TMS derivatives are either unstable[12] or produce inconsistent quantitative results[2]. The method described in this paper has been in constant use for the past 3 years and thousands of aqueous commercial solutions have been chromatographed.

The acceptance of this method by the South African sugar industry has been realised by ensuring careful attention to the analytical detail. A paper describing the routine procedures and method evaluation techniques should be published shortly[14].

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# RESOLUTION OF TRITIUM-LABELLED AMINO ACID RACEMATES BY LIGAND-EXCHANGE CHROMATOGRAPHY

## II*. L-HYDROXYPROLINE- AND L-PHENYLALANINE-MODIFIED RESINS FOR THE RESOLUTION OF COMMON α-AMINO ACIDS**

Yu. A. ZOLOTAREV*, N. F. MYASOEDOV, V. I. PENKINA, I. N. DOSTOVALOV and O. V. PETRENIK

*Institute of Molecular Genetics, U.S.S.R. Academy of Sciences, Kurchatov Sq. 46, Moscow (U.S.S.R.)*

and

V. A. DAVANKOV

*Institute of Organo-Element Compounds, U.S.S.R. Academy of Sciences, Moscow (U.S.S.R.)*

## SUMMARY

Preparations of racemic multiple tritiated valine, histidine and alanine with high specific activities were resolved into enantiomers using ligand-exchange chromatography on $Cu^{2+}$-saturated L-hydroxyproline-modified polystyrene and L-phenylalanine-modified polyacrylamide. These two resins allow the resolution of all common amino acids on a preparative scale and their optical and radiochemical purity to be established. The method does not require any chemical modification of the racemate to be resolved, does not impose any limitations on its specific activity and provides for the simultaneous radiochemical purification of the enantiomers.

## INTRODUCTION

The range of methods for resolving racemates of α-amino acids into constituent optically active enantiomers is severely limited when labelled compounds of high specific activity are involved. All methods involving crystallization procedures are inapplicable because of rapid radiolysis of the radioactive compound in a condensed phase. Natural enzymes commonly used to modify selectively one of the enantiomers in solution are easily inactivated by radiation. Much more promising are chromatographic methods, particularly ligand-exchange chromatography on chiral chelating resins (for a review, see ref. 1). This chromatographic method does not require any chemical modification of the amino acid that would unavoidably lead to a decreased yield and loss of the radioactive compound.

In Part I[2] we described the resolution of D,L-[$^3$H]valine with a specific activity

* For part I, see ref. 2.

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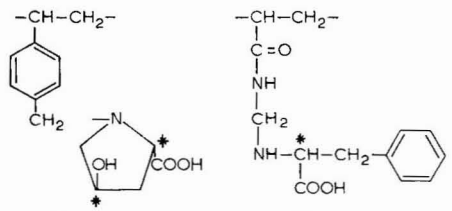
of $1.4 \cdot 10^{12}$ Bq/mmol on a column containing L-hydroxyproline-modified polystyrene resin. This paper is concerned with further optimization of the resolution procedure using this type of resin (I) and the synthesis of an L-phenylalanine-modified polyacrylamide gel (II) which easily resolves racemates of most common amino acids.




I II

## EXPERIMENTAL

### *Sorbents*

The synthesis of sorbent I by interaction of methyl L-hydroxyprolinate with chloromethylated cross-linked polystyrene was described earlier[3]. The starting copolymer contained 0.7% of divinylbenzene and was additionally cross-linked with monochlorodimethyl ether to give a total degree of cross-linking of 6 mol%. The water content of the swollen resin was 250% and the exchange capacity was 3.8 mmol of residues of L-hydroxyproline per gram of sorbent.

Sorbent II was obtained by treatment of Bio-Gel P-4 polyacrylamide beads (Serva, Heidelberg, G.F.R. )with formaldehyde and L-phenylalanine. The sorbent contained 1.4 mmol of groupings of L-phenylalanine per gram. The water uptake was 300%.

Before packing into columns, the sorbents were treated with an excess of copper(II)–ammonia solution and subsequently with a solution of potassium chloride in 1.0 *N* ammonia to achieve the desired content of $Cu^{2+}$ ions in the sorbent.

### *Chromatography of racemates*

The copper-loaded sorbents I and II suspended in 0.1 *N* ammonia or 1% ammonium phosphate solution (pH 9.2), respectively, were slurry-packed into glass columns and conditioned by passing the same eluents through them.

The racemic amino acids were introduced into the top of the column with the help of a micro-syringe. To detect the enantiomers resolved, the Radiochromatograph 2301 chromatographic system (U.S.S.R.) was used, equipped with a flow radioactivity detector cell of volume 170 $\mu$l and made of scintillating quartz. Another detector was a flow photometer operated at 210, 250 or 280 nm.

### *Isolation and characterization of enantiomers*

Using the hydrolytical stable resin of type I and ammonia solutions as the eluent, the resolved amino acid enantiomers can be easily obtained by evaporation of the corresponding eluate fractions, which should be previously purified to remove trace amounts of $Cu^{2+}$. The purification consists in filtering the eluate through a small column (15 × 8 mm I.D.) with ANKB-50 chelating resin (polystyrene bearing iminodiacetate groups).

To obtain directly D- and L-enantiomers of [$^3$H]histidine in a copper-free state, the lower part (20 mm) of the chromatographic column was packed with copper-free resin I and the remainder of the resin (100 mm) was saturated with $Cu^{2+}$ ions to 45% of the theoretical capacity. In contrast to many other amino acids, histidine enantiomers can be detected photometrically without being complexed with $Cu^{2+}$.

With resin II and phosphate-containing eluents, the fractions of resolved enantiomers have to be purified to remove mineral salts. Therefore, the enantiomers were sorbed on the $Cu^{2+}$ form of the ANKB-50 iminodiacetate resin, washed with water and desorbed with 0.3 *N* ammonia solution.

The purity of the isolated enantiomers was tested by thin-layer chromatography (TLC) on Silufol UV-254 plates and by treatment with specific amino acid oxidases in a standard manner.

## RESULTS AND DISCUSSION

When dealing with multiple tritiated amino acids of very high specific radioactivity, chromatography seems to be the method of choice for separating the optical isomers and purifying them simultaneously to remove the contaminating radiolysis products. One of the chiral resins suitable for this purpose is L-hydroxyproline incorporated in a macronet polystyrene with active sites of type I. When saturated with $Cu^{2+}$, this resin displays high enantioselectivity towards optical isomers, which allows one to resolve racemates of several amino acids[4].

In order to shorten the time of exposure of the sorbent to irradiation, measures should be taken to optimize the chromatography process. As gel diffusion is the rate-determining factor in establishing the ligand-exchange equilibrium between the resin phase and solution[2,5], enhancement of the chromatographic efficiency is achieved by using a sorbent of enhanced swellability and small particle size. Figs. 1 and 2 show the results of the preparative chromatographic resolution of enantiomers of DL-[$^3$H]-valine and DL-[$^3$H]histidine under optimized conditions. Here, resin of type I was used with particle diameter 25–32 $\mu$m (irregularly shaped particles). The degree of saturation of the resin with $Cu^{2+}$ and the ammonia concentration in the eluent were selected so as to complete the process in 1–2 h. Fig. 1 clearly indicates the presence

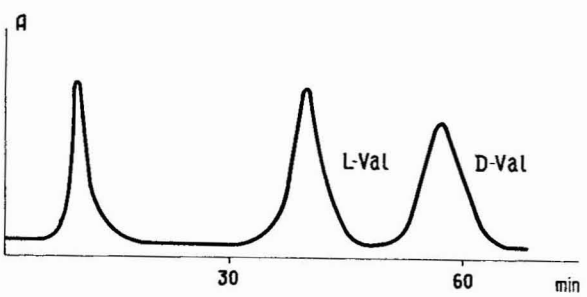



Fig. 1. Chromatography of DL-[$^3$H]valine (40 $\mu$g in 0.1 ml of water; activity $7.4 \cdot 10^8$ Bq; specific activity $2.1 \cdot 10^{12}$ Bq/mmol) on the L-hydroxyproline-containing resin I (particle diameter, $d_p$ = 25–32 $\mu$m; saturation with $Cu^{2+}$ 70%). Column, 300 × 4 mm I.D.; eluent, 0.15 *N* ammonia solution; flow-rate, 16 ml/h.

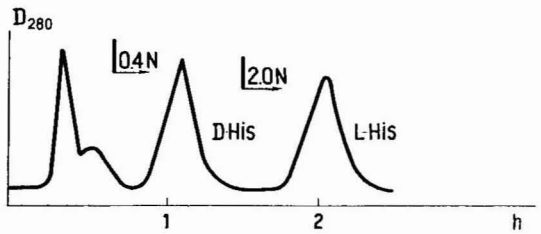



Fig. 2. Chromatography of DL-[$^3$H]histidine (500 $\mu$g in 0.1 ml of water; activity $7.4 \cdot 10^9$ Bq; specific activity $2.4 \cdot 10^{12}$ Bq/mmol) on the L-hydroproline-containing resin I ($d_p$ = 25–32 $\mu$m; saturation with $Cu^{2+}$ 45%). Column, 120 × 8 mm I.D. (lower 20 mm of the resin bed free of copper); eluent, 0.1, 0.4 and 2.0 *N* ammonia solution; flow-rate, 40 ml/h.

of radioactive contaminants in the starting racemate and the efficiency of the radiochemical purification of the enantiomers.

Fig. 3 demonstrates the analytical possibilities opened up by the ligand-exchange chromatography of racemates. The enantiomeric composition of an amino acid sample can be determined within 15–20 min on a 10-cm column of 2 mm I.D. filled with *ca.* 10-$\mu$m particles of resin I. Another approach for evaluating both the optical and radiochemical purity of the resolved enantiomers is the chromatography of, *e.g.*, $3.7 \cdot 10^7$ Bq of a labelled L-[$^3$H]amino acid in the presence of 1 mg of DL-amino acid. The distribution of radioactivity between the L- and D-fractions reflects the optical purity of the labelled product, and the total radioactivity yield in the two amino acid fractions represents its radiochemical purity. According to this test, the optical purity of enantiomers obtained by the proposed preparative resolution is not less than 99%. This is in agreement with the results of standard tests using specific amino acid oxidases. The radiochemical purity of the enantiomers exceeds 95% according to both the above chromatographic method and the standard method using TLC on Silufol UV-254 with different eluting mixtures.

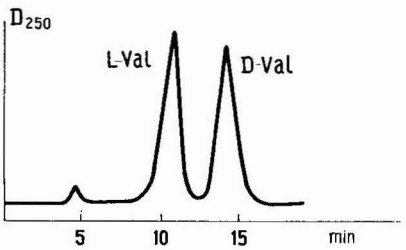



Fig. 3. Chromatography of DL-valine (15 $\mu$g) on the L-hydroproline-containing resin I ($d_p \approx 10$ $\mu$m; saturation with $Cu^{2+}$ 70%). Column, 100 × 2 mm I.D.; eluent, 0.25 *N* ammonia solution; flow-rate, 5 ml/h; pressure, 20 bar.

Although easy to use, the L-hydroxyproline-containing polystyrene resin I displays insufficient enantioselectivity in the chromatography of aspartic and glutamic acids, asparagine, ornithine, lysine, methionine, alanine and $\alpha$-aminobutyric acid[4]. These racemates, and many other amino acids, can best be resolved by using sorbent II obtained by treatment of Bio-Gel P-4 beads (particle diameter <64 $\mu$m) with formaldehyde and L-phenylalanine. This type of sorbent has been mentioned

elsewhere[6], but no enantioselectivity was found in resolution tests with proline and valine. In our experiments, sorbent II showed enantioselectivity of at least $\alpha = 1.25$ in all systems tested (see Table I) and a high enough efficiency to resolve all common amino acids. L-Enantiomers of amino acids are always eluated ahead of the D-isomers.

TABLE I

PARAMETERS OF AMINO ACID ELUTION ON THE L-PHENYLALANINE-CONTAINING POLYACRYLAMIDE RESIN II SATURATED WITH $Cu^{2+}$ IONS TO 60%

Eluent, 2% ammonium phosphate solution, pH 9.2. $k'$ = Capacity factor; $\alpha$ = separation factor.

| *Amino acid* | $k'_L$ | $k'_D$ | $\alpha$ |
|---|---|---|---|
| Aspartic acid | 1.02 | 1.34 | 1.31 |
| Glutamic acid | 1.13 | 1.50 | 1.32 |
| Asparagine | 3.04 | 4.12 | 1.35 |
| Glutamine | 1.47 | 2.20 | 1.50 |
| Ornithine | 2.84 | 3.78 | 1.33 |
| Lysine | 6.85 | 9.34 | 1.36 |
| Serine | 2.04 | 2.67 | 1.31 |
| Threonine | 2.52 | 3.36 | 1.33 |
| Methionine | 3.15 | 4.98 | 1.58 |
| Alanine | 1.68 | 2.31 | 1.36 |
| Valine | 1.53 | 2.35 | 1.55 |
| Leucine | 2.29 | 3.26 | 1.42 |
| Norleucine | 2.33 | 3.68 | 1.58 |
| Isoleucine | 1.74 | 2.79 | 1.60 |
| Proline | 3.19 | 5.33 | 1.65 |
| *allo*-Hydroxyproline | 5.25 | 6.59 | 1.25 |
| Tyrosine | 5.17 | 7.58 | 1.37 |
| Phenylglycine | 1.51 | 2.54 | 1.66 |
| Phenylalanine | 3.61 | 4.85 | 1.34 |
| Tryptophan | 8.95 | 12.7 | 1.42 |

Figs. 4 and 5 demonstrate the resolutions of racemic lysine and methionine and Fig. 6 the preparative resolution of DL-[$^3$H]alanine. The resolution of DL-[$^3$H]-glutamic acid on sorbent II was described earlier[7]. In all experiments with sorbent II, ammonium phosphate solution of pH 9.2 was used as the eluent. The purification of the resolved enantiomers to remove mineral salts and trace amounts of $Cu^{2+}$ was effected using chelating resins saturated with $Cu^{2+}$ and free of copper, respectively, as is described under Experimental.

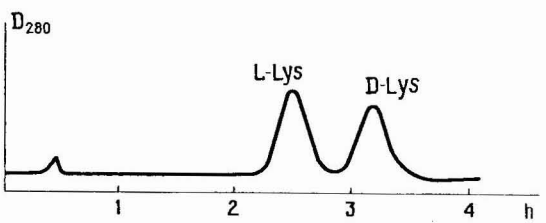



Fig. 4. Chromatography of DL-lysine (300 $\mu$g) on the L-phenylalanine-containing resin II ($d_p \leqslant 64$ $\mu$m; saturation with $Cu^{2+}$ 60%). Column, 300 × 9 mm I.D.; eluent, 2% ammonium phosphate solution, pH 9.2; flow-rate, 30 ml/h.

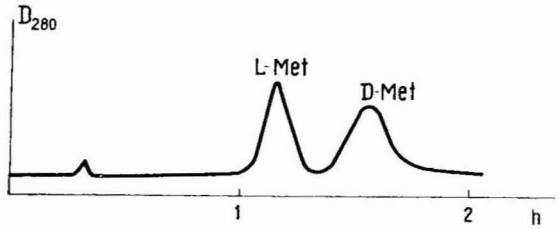



Fig. 5. Chromatography of DL-methionine (300 $\mu$g) on the L-phenylalanine-containing resin II ($d_p$ < 64 $\mu$m; saturation with $Cu^{2+}$ 70%). Column, 190 × 8 mm I.D.; eluent, 2.5% ammonium phosphate solution, pH 9.2; flow-rate, 25 ml/h.

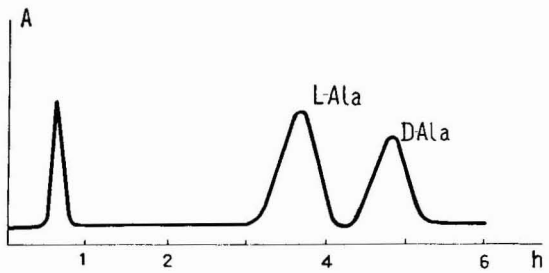



Fig. 6. Chromatography of DL-[$^3$H]alanine (315 $\mu$g in 0.1 ml of water; activity $5.5 \cdot 10^9$ Bq; specific activity $1.7 \cdot 10^{12}$ Bq/mmol) on the L-phenylalanine-containing resin II ($d_p$ < 64 $\mu$m; saturation with $Cu^{2+}$ 70%). Column, 300 × 9 mm I.D.; eluent, 0.1% ammonium phosphate solution, pH 9.2; flow-rate, 25 ml/h.

## CONCLUSION

The reliability and efficiency of the ligand-exchange chromatographic resolution of racemates make it possible to obtain optically and radiochemically pure amino acids from a racemic stock solution shortly before they are required for use.

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# RAPID METHOD FOR THE DETERMINATION OF TETRAALKYLTIN COMPOUNDS IN VARIOUS KINDS OF BIOLOGICAL MATERIAL BY GAS CHROMATOGRAPHY

YASUAKI ARAKAWA*

*Department of Hygiene and Preventive Medicine, Faculty of Medicine, University of Tokyo, Tokyo (Japan)*

OSAMU WADA

*Gunma University, 7-3-1, Hongo, Bunkyo-ku, Tokyo 113 (Japan)*

T. H. YU

*Department of Hygiene and Preventive Medicine, Faculty of Medicine, University of Tokyo, Tokyo (Japan)*

and

HIDEAKI IWAI

*Gumna University, 7-3-1, Hongo, Bunkyo-ku, Tokyo 113 (Japan)*

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## SUMMARY

A rapid gas chromatographic method is described for the simultaneous determination of tetraalkyltin compounds in biological materials. Tetraalkyltins were rapidly purified by direct passage through a silica gel column after extraction from the homogenized tissues with *n*-hexane. Gas chromatographic analysis was alternatively carried out with PEG 20M at temperatures from 50 to 150°C. A hydrogen flame-ionization detector was more sensitive and selective towards tetraakyltins than an electron-capture detector. Detection limits reached $1 \cdot 10^{-8}$ g for tetraalkyltins. Recoveries of tetraalkyltins added to various tissues at the 85-nmole level ranged from 97 to 104%. *In vivo* studies indicated that for a sample containing more than 0.1 μg of tetraalkyltins per gram of tissue, the proposed method is accurate enough for quantitative analysis.

## INTRODUCTION

Organotin compounds have been widely used as polymer stabilizers, fungicides, insecticides, organic catalysts, oil additives, etc. In recent years, there has been concern over the potential health danger from these compounds. Toxicological reports[1–4] have confirmed that tetraalkyltins produce the same effect in animals as trialkyltins, which are the most toxic organotin compounds towards the central nervous system. Therefore, it is important that sensitive and accurate methods are

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established for the determination of official tolerance limits of tetraalkyltin and other organotin compounds. In addition, such methods should also be simple and rapid and capable of determining organotin compounds in various kinds of biological material.

Numerous methods have been published for the determination of organotin compounds. However, most of the methods described for the determination of tetraalkyltins are incidental to those of tri-, di- or monoalkyltins. These include the determination of elements in organotin compounds such as tin[5–20], carbon and hydrogen[21,22], halogens, nitrogen and sulphur, and the determination of organotin compounds themselves[23–44]. The determinations of tin in organotin compounds are based on their conversion into tin(II) and tin(IV) oxides by oxidation with various oxidizing agents, and have been conducted by titrimetric[5,6], complexometric[6–11], spectrometric[12,13], gravimetric[9,14], volumetric[5,15,16], photometric[5,17], X-ray fluorescence[18], X-ray spectrophotometric[19] and polarographic[20] methods after destruction of the organotin compounds. For the determination of organotin compounds themselves, paper chromatographic[23,24], thin-layer chromatographic[25,26], ultraviolet and infrared spectrophotometric[27], nuclear magnetic resonance spectrometric[27,28] and gas chromatographic[29–44] methods have been developed.

Some of these methods, however, are too complicated and others suffer from unsatisfactory sensitivity, precision, reproducibility and specificity and are therefore unsuitable for application to the determination of tetraalkyltins in biological materials. Of the available methods, gas chromatography appears to be the most versatile and applicable to the determination of tetraalkyltins in mammals.

In this work, we examined the gas chromatographic separation of tetraalkyltins and the purification of tetraalkyltins from biological materials, and established a rapid procedure for the simultaneous determination of tetraalkyltins in various kinds of biological materials by gas chromatography.

## EXPERIMENTAL

### *Reagents*

Tetraethyltin ($Et_4Sn$), tetrapropyltin ($Pr_4Sn$), tetrabutyltin ($Bu_4Sn$) and tetraethyllead ($Et_4Pb$) were obtained from Aldrich (Milwaukee, WI, U.S.A.). The purity of these compounds was not less than 98%. When not of acceptable purity, the compounds were purified by distillation or by silica gel column chromatography (see Fig. 1). Other reagents included special-grade materials and organic solvents, such as silica gel (No. IIA, 100–200 mesh, obtained from Nakarai Chemical, Tokyo, Japan) and *n*-hexane (provided by Wako, Tokyo, Japan).

### *Gas chromatography*

The instrument was a Shimadzu Model GC-6AM gas chromatograph equipped with a hydrogen flame-ionization detector (HFID). A glass tube (200 cm × 3 mm I.D.) was packed with 10% PEG 20M on Shimalite W (80–100 mesh) support. Other gas chromatographic conditions are given in the figures.

### *Preparation of tetraalkyltin compounds from tissues*

The procedure for the preparation of samples for analysis of tetraalkyltin com-

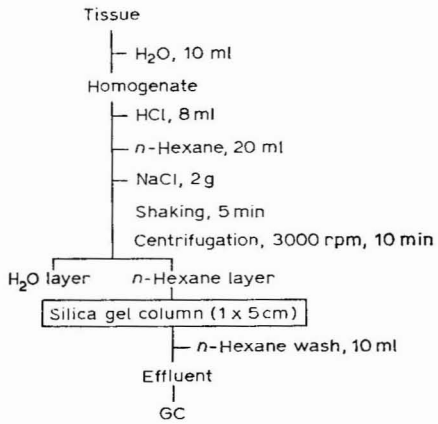


Fig. 1. Preparation of tetraalkyltin compounds from tissues.

pounds in tissues is shown in Fig. 1. A sample of tissue weighing between 1.0 and 5.0 g (wet weight) was homogenized in 10 ml of water. Concentrated hydrochloric acid (8 ml) was carefully added to the homogenate and the contents of the tube was mixed thoroughly and allowed to stand for 5 min. *n*-Hexane (20 ml), sodium chloride (2 g) and a suitable amount of $Et_4Pb$ as internal standard were added and the tetraalkyltins were extracted by shaking for 5 min. After centrifuging for 10 min at 3000 rpm (1000 *g*), the upper *n*-hexane layer was transferred to another flask. The extraction procedure was repeated twice. The *n*-hexane layers were combined and passed directly through a silica gel column (1 × 5 cm, conditioned by washing with *n*-hexane), then washed with 10 ml of *n*-hexane. The effluent was collected in a 50-ml pear-shaped flask and concentrated to an appropriate volume under reduced pressure at about 20°C. A volume of 1–2 $\mu$l of the concentrated solution was injected directly into the gas chromatograph under suitable conditions.

*Animals*

Randomized groups of five to eight mature male rabbits (Japanese White rabbit, 3.5–4.0 months old, 2.0–2.3 kg body weight, obtained from Nippon Bio-supp. Centre, Tokyo, Japan) were used.

*Administration of tetraalkyltins*

For intravenous administration, tetraalkyltin homologues were first dissolved in 100% ethanol, then were carefully mixed with saline solution in a syringe (1 part of ethanol to 2–3 parts of saline solution). The final concentration of ethanol in the preparation was 20–30%. The preparation was slowly injected into the ear vein of rabbits, the dose levels used being 2.0 mg/kg body weight for tetraethyltin, 2.5 mg/kg for tetrapropyltin and 3.0 mg/kg for tetrabutyltin (equivalent to 8.5 $\mu$mole/kg of each tetraalkyltin). The rabbits were killed at 30 or 180 min after administration and liver, kidney, brain and whole-blood samples were prepared for gas chromatographic analysis of tetraalkyltins.

## RESULTS AND DISCUSSION

### *Selection of analytical conditions*

*Gas chromatographic conditions.* Using the HFID and an electron-capture detector (ECD), the resolution of tetraalkyltins was examined on various stationary liquid phases and retention times, separating state, peak sharpness and sensitivities were established. The HFID was more sensitive and selective towards tetraalkyltins than the ECD. It was possible to elute tetraalkyltins through polar stationary phases such as polyethylene glycol. In particular, the complete separation of tetraalkyltins was achieved on a 10% PEG 20M column within 25 min at temperatures from 50 to 150°C (Fig. 2). This column also gave satisfactory peak shapes and sensitivity. Low-polarity and non-polar stationary phases such as QF-1, SE-52, SE-30, OV-1, OV-17 and squalane could not be used because of adsorption and decomposition of the tetraalkyltins. The retention time of tetraalkyltins was affected by the polarity of the stationary phase. With polar stationary phases, tetraalkyltins were separated according to their molecular weights and boiling points. The solid support (Shimalite W, 80–100 mesh) used was treated by baking it at 300°C for 5 h, washing it with acid and alkali, drying it at 50°C and silylating it with dimethyldichlorosilane. The air, hydrogen and nitrogen flow-rates were 70, 90 and 60 ml/min, respectively.

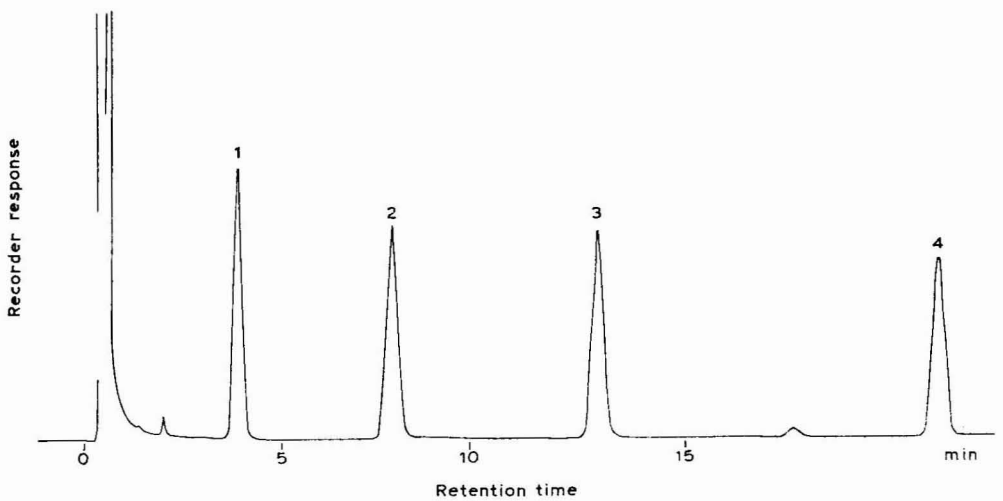


Fig. 2. Gas chromatogram of tetraalkyltin compounds. Column: 10% PEG 20M on Shimalite W (80–100 mesh), 2.0 m × 3 mm I.D. Temperatures: column, programmed from 50 to 150°C at 4°C/min; HFID, 180°C. Flow-rates: air, 70 ml/min; $H_2$, 90 ml/min; $N_2$, 60 ml/min. Sensitivity: 100. Range: 0.8 V. Peaks: 1 = $Et_4Sn$; 2 = $Et_4Pb$ (internal standard); 3 = $Pr_4Sn$; 4 = $Bu_4Sn$.

*Internal standard.* The internal standard should have a retention time at about the mid-point of the chromatogram or in this instance an an elution temperature of about 90–100°C. To minimize errors due to mechanical losses, the internal standard was added to the crude sample before the preparation steps were begun. Tetraethyllead seemed to possess the necessary characteristics and hence was selected as the internal standard.

*Calibration graphs.* Standard solutions containing approximately equal concentrations (about 10 μg/ml) of tetraethyllead and varying concentrations (about 5–20 μg/ml) of the standard tetraalkyltin compounds in *n*-hexane were prepared. Under the gas chromatographic conditions specified in the legend to Fig. 2, calibration graphs were established for peak heights of tetraethyltin, tetrapropyltin and tetrabutyltin. Linear calibration graphs indicated good working ranges for the compounds tested. Detection limits reached $1 \cdot 10^{-8}$ g for tetraalkyltin compounds.

*Sample preparation.* Extraction of tetraalkyltins from tissues with various solvents was examined. Tetraalkyltins with a high solubility in organic solvents could be easily extracted by using low-polarity and non-polar solvents such as benzene, toluene, *n*-hexane and ethyl acetate. *n*-Hexane was selected as the most suitable solvent for the extraction of tetraalkyltins because it was also suitable for the next step, silica gel column chromatography. The recovery of double extractions with *n*-hexane was about 98%.

For the purification of tetraalkyltins from *n*-hexane-soluble substances in biological materials, column chromatography using silica gel was examined. Tetraalkyltins were not adsorbed on *n*-hexane-conditioned silica gel and could be easily separated from other slightly polar substances. Mono-, di- and trialkyltin compounds were adsorbed.

*Analysis of tetraalkyltin mixtures*

Standard solutions containing various amounts of tetraalkyltins in *n*-hexane were analysed according to the proposed procedure. The overall recovery of tetraalkyltins was 98–102% (Table I).

TABLE I

ANALYSIS OF STANDARD SAMPLE

Five standard solutions of tetraalkyltins were subjected to the gas chromatographic method using 10 μg of tetraethyllead as internal standard. Gas chromatographic conditions are described in the legend of Fig. 2.

| *Compound* | *Added (μg)* | *Found (μg)* | | | | | *Average* | |
|---|---|---|---|---|---|---|---|---|
| | | *1* | *2* | *3* | *4* | *5* | *μg* | *Recovery (%)* |
| Tetraethyltin | 10 | 10.2 | 9.8 | 11.3 | 9.5 | 10.0 | 10.2 | 102 |
| Tetrapropyltin | 15 | 14.8 | 15.1 | 15.0 | 15.4 | 15.2 | 15.1 | 101 |
| Tetrabutyltin | 20 | 19.8 | 20.0 | 19.8 | 19.5 | 18.9 | 19.6 | 98 |

*Addition studies*

The application of the method to the analysis of tetraalkyltins in mammals was studied by conducting recovery tests on animal tissues. Equal amounts (85 nmole) of tetraalkyltins were added to various rabbit tissues and the recoveries were determined (Table II). The average recovery was 97–104%. There was no difference in the recoveries from different organs.

*Application to in vivo studies*

Rabbits given tetraalkyltins (8.5 μmole/kg of each) intravenously were killed 30 or 180 min after administration, and the concentrations of tetraalkyltins in the

TABLE II

RECOVERY OF TETRAALKYLTIN COMPOUNDS ADDED TO RABBIT TISSUES *IN VITRO*

Three tetraalkyltins (85 nmole of each) were added to various tissues (5 g) and subjected to the gas chromatographic method using 20 $\mu$g of tetraethyllead as internal standard. Gas chromatographic conditions are described in the legend of Fig. 2. Each result is the average of five determinations (mean $\pm$ standard error).

| *Compound* | *Added ($\mu$g)* | *Organ* | *Average* | |
|---|---|---|---|---|
| | | | *Found ($\mu$g)* | *Recovery (%)* |
| $Et_4Sn$ | 20 | Blood | 20.8 $\pm$ 0.2 | 104.0 $\pm$ 0.9 |
| | | Liver | 20.1 $\pm$ 0.3 | 100.4 $\pm$ 1.5 |
| | | Kidney | 20.4 $\pm$ 0.3 | 101.8 $\pm$ 1.5 |
| | | Brain | 20.6 $\pm$ 0.3 | 103.0 $\pm$ 1.4 |
| $Pr_4Sn$ | 25 | Blood | 25.6 $\pm$ 0.2 | 102.2 $\pm$ 0.9 |
| | | Liver | 25.6 $\pm$ 0.3 | 102.6 $\pm$ 1.1 |
| | | Kidney | 25.5 $\pm$ 0.3 | 102.2 $\pm$ 1.3 |
| | | Brain | 25.5 $\pm$ 0.2 | 101.9 $\pm$ 0.8 |
| $Bu_4Sn$ | 30 | Blood | 29.0 $\pm$ 0.3 | 96.8 $\pm$ 1.1 |
| | | Liver | 30.0 $\pm$ 0.4 | 100.1 $\pm$ 1.2 |
| | | Kidney | 29.8 $\pm$ 0.4 | 99.3 $\pm$ 1.4 |
| | | Brain | 29.9 $\pm$ 0.2 | 99.8 $\pm$ 0.7 |

liver, kidney, brain and whole blood were determined. The results are shown in Table III and Fig. 3. This experiment indicated that for a sample containing more than 0.1 $\mu$g of tetraalkyltins per gram of tissue (wet weight), the method is accurate enough for quantitative analysis.

TABLE III

DISTRIBUTION OF TETRAALKYLTIN COMPOUNDS IN RABBIT ORGANS AFTER INTRAVENOUS ADMINISTRATION

Tissue samples (1–5 g) from rabbits given three tetraalkyltins (8.5 $\mu$mole/kg of each) were subjected to the gas chromatographic method using 10 $\mu$g of tetraethyllead as internal standard. Gas chromatographic conditions are described in the legend of Fig. 2. Tetraalkyltins are expressed as $\mu$g/g of tissue (wet weight). Results are means $\pm$ standard errors (5–8 animals per group).

| *Organ* | *Compound* | *Time after administration (min)* | |
|---|---|---|---|
| | | *30* | *180* |
| Blood | $Et_4Sn$ | 12.3 $\pm$ 0.25 | 7.3 $\pm$ 0.10 |
| | $Pr_4Sn$ | 13.2 $\pm$ 0.23 | 2.4 $\pm$ 0.04 |
| | $Bu_4Sn$ | 12.5 $\pm$ 0.25 | 2.3 $\pm$ 0.04 |
| Liver | $Et_4Sn$ | 5.5 $\pm$ 0.07 | 2.0 $\pm$ 0.02 |
| | $Pr_4Sn$ | 1.5 $\pm$ 0.02 | 7.5 $\pm$ 0.17 |
| | $Bu_4Sn$ | 1.2 $\pm$ 0.02 | 7.8 $\pm$ 0.15 |
| Kidney | $Et_4Sn$ | 3.7 $\pm$ 0.05 | 6.4 $\pm$ 0.12 |
| | $Pr_4Sn$ | 3.9 $\pm$ 0.05 | 1.6 $\pm$ 0.03 |
| | $Bu_4Sn$ | 2.7 $\pm$ 0.03 | 1.0 $\pm$ 0.03 |
| Brain | $Et_4Sn$ | 1.2 $\pm$ 0.02 | 0.5 $\pm$ 0.01 |
| | $Pr_4Sn$ | 0.5 $\pm$ 0.01 | 0.6 $\pm$ 0.01 |
| | $Bu_4Sn$ | 0.5 $\pm$ 0.01 | 0.6 $\pm$ 0.02 |

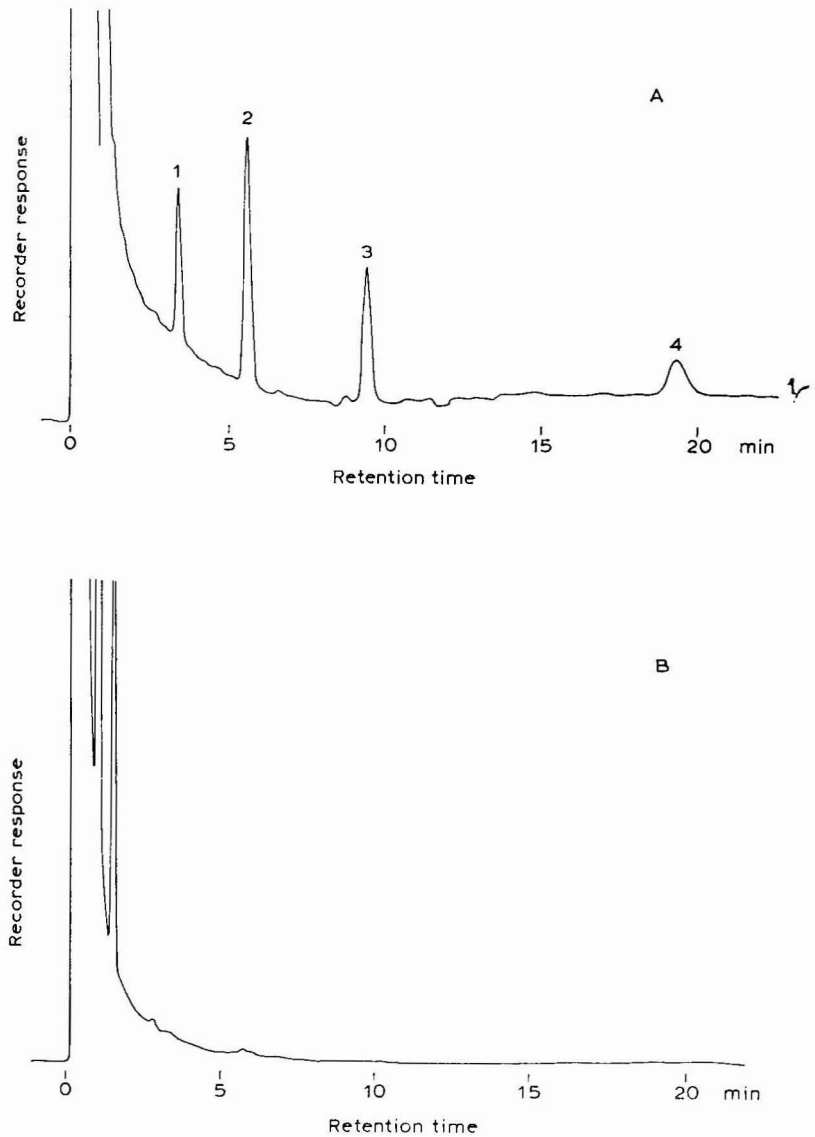


Fig. 3. Gas chromatograms of the liver extracts of rabbit (A) treated with tetraalkyltin compounds and (b) untreated. Column: 10% PEG 20M on Shimalite W (80–100 mesh), 2.0 m × 3 mm I.D. Temperatures: column, programmed from 70 to 130°C at 4°C/min; HFID, 180°C. Carrier gas: $N_2$ at 90 ml/min. Sensitivity: 100. Range: 0.8 V. Peaks: 1 = $Et_4Sn$; 2 = $Et_4Pb$ (internal standard;) 3 = $Pr_4Sn$; 4 = $Bu_4Sn$.

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## Note

# Effect of degree of coating on column efficiency in liquid chromatography

YOUNG HWAN KIM* and ARYE TISHBEE
*Department of Organic Chemistry, The Weizmann Institute of Science, Rehovot (Israel)*
(Received November 3rd, 1980)

Silica gel which is physically coated with a solid substance is still a useful stationary phase in liquid chromatography (LC), in spite of the high performance of other LC systems. Such coated systems have been shown to be advantageous for the separation of geometric[1] and optical isomers[2,3]; transition-metal complexes[4,5] and charge-transfer acceptors for complexation with polyaromatic hydrocarbons (PAHs)[1–3] are typical examples.

*In situ*[3,4] coating is a generally accepted method, but this technique possesses intrinsic problems, such as inhomogeneity of the coating and uncertainty of the amount of the coating material. In the course of high-performance liquid chromatography studies on charge-transfer complexation between biological compounds with PAHs we observed that the amount of coating on silica gel seriously affects the column performance.

We report here the influence of the amount of riboflavin (vitamin $B_2$), the coating material, on silica gel for PAH separation.

## EXPERIMENTAL

The experimental procedure used for the preparation of the high-performance column has been reported[6]. Since the purpose of this study was to examine the effect of the amount of coating on the performance of the column and not to optimize the conditions, an inexpensive silica gel was employed. A known amount of Partisil 20 (Whatman, Maidstone, Great Britain) was added to an aqueous solution containing a known amount of riboflavin. Water was evaporated slowly at 80°C using a rotary evaporator. Coating was complete in 2–3 h. The coated silica gel was finally dried in high vacuum (1.0 mmHg) at 80°C overnight. A 25 × 0.21 cm I.D. stainless-steel column was prepared by the dry-packing method. In order to ensure comparable conditions, the same stainless-steel tube was used for the different stationary phases. A Waters 6000 pump, Reodyne 2710 injector, and LDC UV detector (254 mm) were employed.

## RESULTS AND DISCUSSION

Naphthalene, phenanthrene, and pyrene were taken as a standard mixture in methylene chloride solution. Silica gel columns which were coated with 0%, 2%, 5%,

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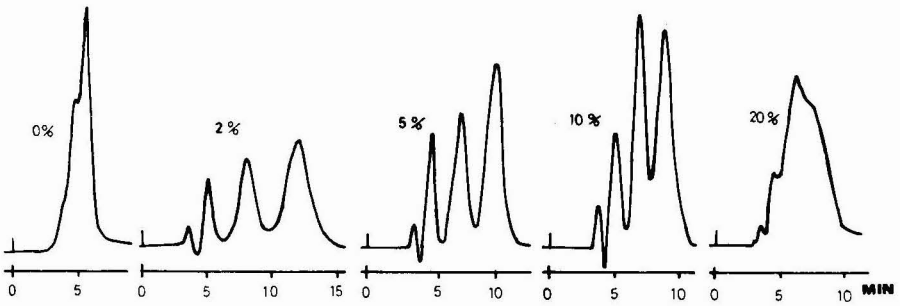


Fig. 1. Separation of PAHs on a riboflavin-coated column. The peaks are naphthalene, phenanthrene and pyrene, in order of increasing retention time. Percentages above the chromatogram indicate coating amount. Column dimensions, 25 × 0.21 cm I.D.; eluent, 10% $CH_2Cl_2$–*n*-hexane; flow-rate, 0.5 ml/min.

10% and 20% (w/w) riboflavin were prepared. The chromatograms of the standard mixture on these columns are shown in Fig. 1. The striking effect of riboflavin on the separation can be seen by comparing the chromatogram obtained with the uncoated silica gel column and the 2% coated column. The column performance starts to deteriorate when the amount of coating material is more than 5%. The retention time also changes according to the coating amount.

The effective molecular area of riboflavin on silica gel was calculated to be 7.4 $nm^2$, according to Snyder[7]. Assuming the surface area of the silica gel to be 400 $m^2/g$, *ca.* 3.3% coating should provide a monolayer coating. Apparently, exceeding this amount does not contribute appreciably to the retention, but it disturbs mass transfer by blocking the pores of the silica gel. Furthermore, the surface area of the coated silica gel is decreased when the coating material is coated as a multilayer. This argument may explain why both performance and retention decrease with increasing amounts of coating material on the silica gel. It seems, however, that since a true monolayer coating is difficult to achieve, the reproducibility of the column in this range is problematic. For column reproducibility, probably somewhat more than a monolayer coating is preferential, as long as the column performance is satisfactory.

This study suggests that when coated silica gel is employed for separation, the surface area and pore size of the silica gel, as well as the effective molecular size of coating material, dictate the amount of coating required for optimal separation conditions.

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CHROM. 13,498

## Note

# Retention behavior of selected colchicine derivatives on reversed-phase high-performance liquid chromatographic systems

A. E. KLEIN and P. J. DAVIS*

*Division of Medicinal Chemistry and Pharmacognosy, College of Pharmacy, The University of Texas at Austin, Austin, TX 78712 (U.S.A.)*

(Received November 3rd, 1980)

Colchicine (I), the major alkaloid of *Colchicum* species, is used in the treatment of gout, and, together with colchicine derivatives, are of interest as potential antineoplastic agents[1]. We are currently examining the use of microorganisms to prepare metabolically derivatives of colchicine[2]. Additionally, we are examining colchiceine (II) and its derivatives as potential microbial metabolites of colchicine[3–5].

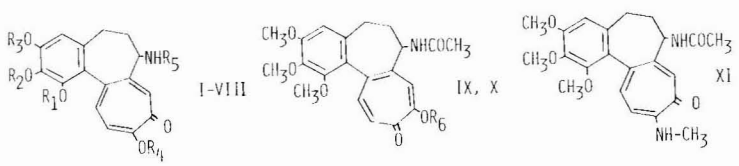


Fig. 1. Structural formulas of colchicines and colchiceine. I: $R_1$ to $R_4 = CH_3$, $R_5 = COCH_3$; II: $R_1$ to $R_3 = CH_3$, $R_4 = H$, $R_5 = COCH_3$; III: $R_1$ to $R_4 = CH_3$, $R_5 = H$; IV: $R_1$ to $R_5 = CH_3$; V: $R_1$, $R_2$, $R_4 = CH_3$, $R_3 = H$, $R_5 = COCH_3$; VI: $R_1$, $R_3$, $R_4 = CH_3$, $R_2 = H$, $R_3 = COCH_3$; VII: $R_2$, $R_3$, $R_4 = CH_3$, $R_1 = H$, $R_5 = COCH_3$; VIII: $R_1$ to $R_3 = CH_3$, $R_4 = C_2H_5$, $R_5 = COCH_3$; IX: $R_6 = CH_3$; X: $R_6 = C_2H_5$.

In order to analyze and quantify colchicine and five colchicine derivatives in microbiological systems, we have recently described a selective high-performance liquid chromatographic (HPLC) procedure[6]. We have also reported a derivatization technique that enables colchiceine (II) to be determined in the presence of colchicine (I) by HPLC[7]. In complex mixtures, it was discovered that certain derivatives of I co-chromatographed, and further studies were initiated to determine the retention behavior of a variety of colchicines with different reversed-phase HPLC systems. In the present work the retention behavior of colchicine and nine of its derivatives was evaluated for ternary mobile phase systems (*i.e.* acetonitrile–methanol–buffer) of varying concentration. The derivatives studied include the N-desacetylcolchicine (III), N-desacetyl-N-methylcolchicine (*i.e.* demecolcine) (IV), 3-demethylcolchicine (V), 2-demethylcolchicine (VI), 1-demethylcolchicine (VII), ethylcolchicinate (VIII), isocolchicine (IX), ethylisocolchicinate (X), and N-methylcolchiceinamide (XI). Four

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reversed-phase columns were compared for their relative retention behavior and conclusions were drawn as to the most suitable HPLC system for rapid identification of these compounds.

## EXPERIMENTAL

### *HPLC system*

A Model 950 pump and 970A variable-wavelength detector (Tracor, Austin, TX, U.S.A.) with a Model 7120 100-$\mu$l loop injector (Rheodyne, Berkeley, CA, U.S.A.) were employed for all analyses. Detection was at 350 nm, and a Model HP-3380A reporting integrator (Hewlett-Packard, Palo Alto, CA, U.S.A.) at an input sensitivity of 0.1 V/a.u. and a slope sensitivity of 1 mV/min was used for peak area measurement and chromatogram recording. The flow-rate was constant at 2.0 or 3.0 ml/min. $\mu$Bondapak $C_{18}$ and phenyl columns were obtained from Waters Assoc. (Milford, MA, U.S.A.) and LiChrosorb RP-18 and RP-8 columns from Alltech (Arlington Heights, IL, U.S.A.) Dead time ($t_0$) was measured by the pressure fluctuation observed on the baseline after an injection of mobile phase. Analyses were performed with methanol–acetonitrile–phosphate buffer mobile phases of varying composition.

### *Reagents*

Organic solvents used in the mobile phase were chromatographic quality (LiChrosorb; MCB, Cincinnati, OH, U.S.A.). Water was deionized and doubly distilled in glass. Mobile phases were prepared by filtering individual solvents through glass fiber pads, GF/F grade (Whatman, Clifton, NJ, U.S.A.) mixing and degassing by sonication prior to use.

### *Standard compounds*

Colchicine (I) and N-methylcolchiceinamide (XI) were purchased from Aldrich (Milwaukee, WI, U.S.A.). Colchiceine (II) was prepared as described[7] by the mild acid treatment of colchicine according to the method of Zeisel[8], and was identical to a sample provided by T. J. Fitzgerald of Florida A. & M. University (Tallahassee, FL, U.S.A.). Ethylisocolchicinate (X) and ethylcolchicinate (VIII) and isocolchicine (IX) were prepared as described in ref. 7.

Samples of colchicine derivatives III–VII were kindly provided by Dr. J. A. R. Mead and Dr. A. Brossi of the National Institutes of Health (Bethesda, MD, U.S.A.). All standard compounds were homogenous as determined by thin-layer chromatography (TLC) and HPLC.

## RESULTS AND DISCUSSION

The retention behavior of colchicine derivatives I and III–XI were studied to select an appropriate HPLC system. Considerable variation in capacity ratios was observed with different reversed-phase packings when operating with our reported mobile phase[6], as shown in Table I. A 30-cm $\mu$Bondapak $C_{18}$ column was able to separate all ten compounds, but the adjusted retention time for the last solute, N-methylcolchiceinamide (XI), was greater than 30 min at a flow-rate of 3 ml/min. By comparison, a shorter 15-cm $\mu$Bondapak–phenyl column eluted this compound more

TABLE I

RETENTION BEHAVIOR OF COLCHICINE DERIVATIVES ON REVERSED-PHASE SILICA GEL COLUMNS

Acetonitrile–methanol–phosphate buffer (pH 6, 0.022 *M*) (16:5:79) as mobile phase. Each packing particle size = 10 $\mu$m.

| *Compound* | | *Capacity ratio (k')* | | | |
|---|---|---|---|---|---|
| | | *Column** | | | |
| | | *A* | *B* | *C* | *D* |
| 3-Demethylcolchicine | (V) | 4.1 | — | 3.6 | 5.4 |
| 2-Demethylcolchicine | (VI) | 4.8 | 3.9 | 4.1 | 6.3 |
| N-Desacetylcolchicine | (III) | 7.1 | — | 6.1 | 10.1 |
| 1-Demethylcolchicine | (VII) | 9.4 | — | 7.9 | 14.8 |
| Demecolcine | (IV) | 10.7 | — | 9.4 | 14.8 |
| Colchicine | (I) | 14.2 | 10.0 | 11.2 | 21.1 |
| Ethylisocolchicinate | (X) | 18.2 | 10.7 | 14.4 | 27.1 |
| Ethylcolchicinate | (VIII) | 26.0 | 15.7 | 19.8 | 43.1 |
| N-Methylcolchiceinamide | (XI) | 38.5 | 21.1 | 27.4 | 60.2 |
| $t_0$ (min) at 3 ml/min flow-rate | | 0.87 | 0.69 | 1.2 | 0.38 |

* A = $\mu$Bondapak $C_{18}$ (30 cm); B = $\mu$Bondapak–phenyl (15 cm); C = LiChrosorb RP-8 (25 cm); D = LiChrosorb RP-18 (10 cm).

quickly, but with some loss in resolution between colchicine and ethylisocolchicinate (I and X). A 25-cm LiChrosorb RP-8 column produced a similar but slightly more time consuming separation for most solutes compared to that with the $\mu$Bondapak $C_{18}$ column, while a 10-cm LiChrosorb RP-18 column was unable to separate 1-demethylcolchicine (VII) from demecolcine (IV) but eluted all solutes within 22 min. This short column was able to separate completely the ethyl isomers (VIII and X) and N-methylcolchiceinamide (XI) within 16 min at a flow-rate of 4 ml/min.

Variations in capacity ratio were also observed when the composition of the mobile phase was changed slightly, as shown for the $\mu$Bondapak $C_{18}$ column in Fig. 2A–C. A change of 1 % in the acetonitrile fraction in the mobile phase produced dramatic changes in the retentions of all of the colchicine derivatives (Fig. 2A), while an equivalent change in the methanol fraction did not alter the capacity ratios as sharply (Fig. 2B). The retentions of the easily ionizable colchicine derivatives III and IV showed major changes as the pH of the phosphate buffer was altered (Fig. 2C), as had previously been noticed[6]. These two compounds were separated most completely from the other compounds at pH 6 as indicated in Fig. 2C and in Fig. 3. The non-ionizable colchicine derivatives showed only slight variations in retention with changes in buffer pH.

The $\mu$Bondapak $C_{18}$ column provided the best separation for all ten colchicine derivatives with the acetonitrile–methanol–phosphate buffer (pH 6) (16:5:79) mobile phase, as shown in Fig. 3, for a flow-rate of 3 ml/min. The N-methylcolchiceinamide peak, which is not included in the figure, eluted at 34.4 min. This system has been chosen for the analysis of colchicine using demecolcine as the internal standard because of its resolving power[7]. Isocolchicine (IX) was the sole colchicine derivative that was not completely resolved from the internal standard with this system. It is

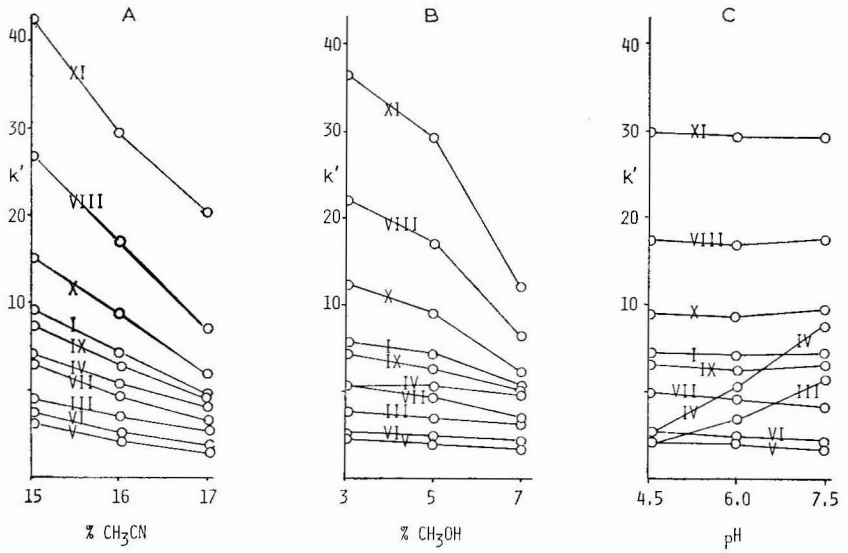


Fig. 2. Capacity ratio ($k'$) as a function of composition of the ternary mobile phase. Compound numbers correspond to those described in Fig. 1. (A), Variation of acetonitrile concentration from 15–17% (phosphate buffer pH 6, 80–78%) with methanol constant at 5%; (B), variation of methanol concentration from 3–7% (phosphate buffer pH 6, 81–77%) with acetonitrile constant at 16%; (C), variation in pH of the phosphate buffer ($\mu = 0.05, 0.022$ $M$ in all cases) in a mobile phase composed of methanol–acetonitrile–buffer (5:16:79).

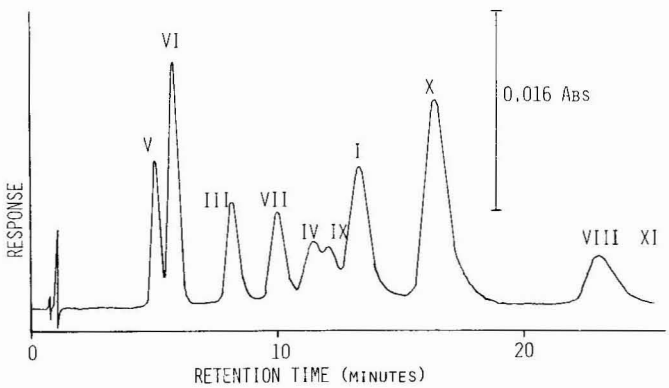


Fig. 3. HPLC separation of colchicine (I) and derivatives (III–X) on a 30-cm $\mu$Bondapak $C_{18}$ column with methanol–acetonitrile–phosphate buffer (pH 6) (5:16:79). The compound numbers correspond to those in Fig. 1.

inconceivable, however, that this compound would be present as a metabolite of colchicine, and also would not be chosen as a potential internal standard. The iso derivatives IX and X eluted before the corresponding normal-colchicines I and VIII on all the reversed-phase packings studied. The increased polarity of the iso series is also consistent with TLC mobility on silica adsorbents[6].

The shorter reversed-phase columns listed in Table I do have utility in specific analyses, and have proved especially useful for the rapid and complete separation of the ethyl derivatives VIII and X from colchicine. The 10-cm LiChrosorb RP-18 column is now being employed for the rapid analysis of colchiceine (II) as its ethylated derivatives (VIII and X) in the presence of colchicine in microbial systems[7]. It is hoped that the systems described herein will aid others in specific applications relating to the HPLC analysis of colchicine and its derivatives.

ACKNOWLEDGEMENTS

This work was supported by U.S. Public Health Service Grant CA-24171. The authors also wish to thank Dr. J. A. R. Mead and Dr. A. Brossi of the National Institutes of Health for generously supplying colchicine derivatives III–VII, and Dr. T. J. Fitzgerald, Florida A. & M. University, for supplying colchiceine (II).

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CHROM. 13,514

## Note

# Gas chromatographic separation of amino acid, amine and carboxylic acid enantiomers with α-hydroxycarboxylic acid esters as chiral stationary phases

NAOBUMI ÔI*, HAJIMU KITAHARA and TADASHI DOI

*Institute for Biological Science, Sumitomo Chemical Co., Ltd., 4-2-1 Takatsukasa, Takarazuka-shi, Hyogo-ken 665 (Japan)*

(Received November 11th, 1980)

The optically active stationary phases used hitherto for the separation of amino acid, amine and carboxylic acid enantiomers by gas chromatography involve NH groups linked to the asymmetric carbon atom, which form diastereomeric hydrogen bonds with solutes. Examples are N-acyl amino acid esters[1], N-acyl dipeptide esters[2] and N-acyl amines[3].

Recently we found that α-hydroxycarboxylic acid ester enantiomers can be resolved on amino acid derivatives[4]. This suggested that some α-hydroxycarboxylic acid esters would be effective as optically active stationary phases and led us to this work.

In this paper we describe the separation of amino acid, amine and carboxylic acid enantiomers with di-*l*-menthyl (+)-tartrate and di-*dl*-menthyl (−)-malate as stationary phases.

## EXPERIMENTAL

Gas chromatography was carried out with a Shimadzu GC-7A gas chromatograph equipped with a flame-ionization detector. Glass capillary columns (40 m × 0.25 mm I.D.) coated with α-hydroxycarboxylic acid esters were used.

Di-*l*-menthyl (+)-tartrate was prepared from (+)-tartaric acid by treatment with *l*-menthol in the presence of concentrated sulphuric acid for several hours at 100°C. The ester was extracted with chloroform and the solution was washed successively with water, 1 *N* hydrochloric acid and water. After drying over sodium sulphate and evaporation, the ester was purified by column chromatography with silica gel and *n*-hexane–ethyl acetate as the eluent. Di-*dl*-menthyl (−)-malate was similarly prepared from (−)-malic acid by treatment with *dl*-menthol. The structures of these esters were confirmed by infrared and nuclear magnetic resonance spectroscopy and microanalysis. Their specific rotations were $[\alpha]_D^{25} = -69°$ ($c = 1.0\%$, chloroform) in di-*l*-menthyl (+)-tartrate and $[\alpha]_D^{25} = -8°$ ($c = 1.2\%$, chloroform) in di-*dl*-menthyl (−)-malate.

(+)-Tartaric acid, (−)-malic acid and *l*- and *dl*-menthol were commercially available. Various racemic amino acids, amines and carboxylic acids shown in Table I were also commercially available. α-Bromo-β,β-dimethylbutyric acid was prepared in our laboratory.

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## RESULTS AND DISCUSSION

The gas chromatographic results are summarized in Table I. Enantiomers of amino acids, amines and carboxylic acids were resolved into their antipodes. Typical gas chromatograms are shown in Figs. 1–3.

TABLE I

GAS CHROMATOGRAPHIC SEPARATION OF AMINO ACID, AMINE AND CARBOXYLIC ACID ENANTIOMERS ON OPTICALLY ACTIVE α-HYDROXYCARBOXYLIC ACID ESTERS

Glass capillary columns, 40 m × 0.25 mm I.D. Column temperature, 100°C. Carrier gas, helium at a flow-rate of 0.7 ml/min. Stationary phases: A, di-*l*-menthyl (+)-tartrate; B, di-*dl*-menthyl (−)-malate.

| *Compound* | *Stationary phase* | *Retention time* (min)* | | *Separation factor, α (second/first)* |
|---|---|---|---|---|
| | | *First peak* | *Second peak* | |
| Amino acids**: | | | | |
| Alanine | A | 22.26 (L) | 22.71 (D) | 1.020 |
| | B | 111.60 (L) | 112.80 (D) | 1.011 |
| Valine | A | 33.54 (L) | 33.97 (D) | 1.013 |
| | B | 191.00 (L) | 192.50 (D) | 1.008 |
| Leucine | A | 67.07 (L) | 68.93 (D) | 1.028 |
| Amines: | | | | |
| α-Phenylethylamine*** | A | 82.34 (−) | 84.39 (+) | 1.025 |
| α-(2,5-Xylyl)ethylamine§ | A | 272.00 (−) | 281.20 (+) | 1.034 |
| α-Phenylpropylamine§ | A | 164.01 (−) | 169.63 (+) | 1.034 |
| Carboxylic acids: | | | | |
| α-Phenylpropionic acid§§ | A | 176.00 (−) | 178.80 (+) | 1.016 |
| α-Bromo-β,β-dimethylbutyric acid§§§ | A | 62.10 (+) | 63.60 (−) | 1.024 |

* Measured from solvent peak.
** Resolved as N-trifluoroacetyl isopropyl ester.
*** Resolved as N-pentafluoropropyl derivatives.
§ Resolved as N-trifluoroacetyl derivatives.
§§ Chromatographed on 20 m × 0.25 mm I.D. glass capillary column using helium at a flow-rate of 1.3 ml/min. Resolved as isopropylamide.
§§§ Resolved as *tert.*-butylamide.

In 1959 Karagounis and Lippold[5] reported the successful of separation of some racemic compounds by gas chromatography with ethyl *d*-tartrate as a chiral stationary phase, but Goldberg and Ross[6] reported that such results could not be reproduced. Berrod *et al.*[7] studied the resolution of some chiral compounds by gas chromatography on chiral stationary phases derived from (+)-tartaric acid, such as (+)-dodecyl tartrate, and achieved a partial separation of the enentiomers of some alcohols by measuring the optical activities of trapped fractions at the beginning and end of the peak corresponding to the racemic compounds, but the separation was insufficient to observe the commencement of resolution.

To our knowledge this is the first successful gas chromatographic separation of racemic compounds with α-hydroxycarboxylic acid esters, which possess OH

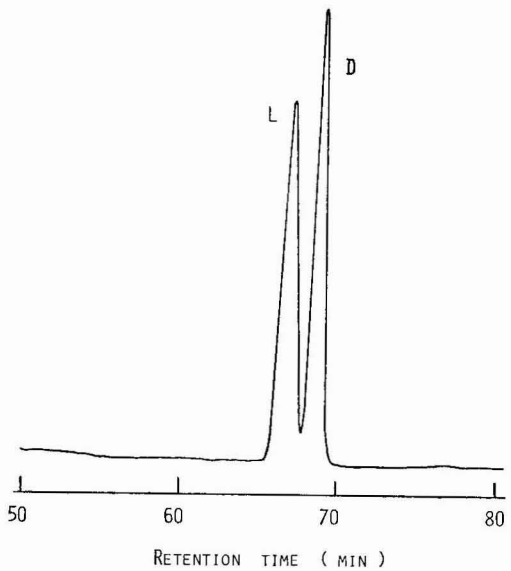
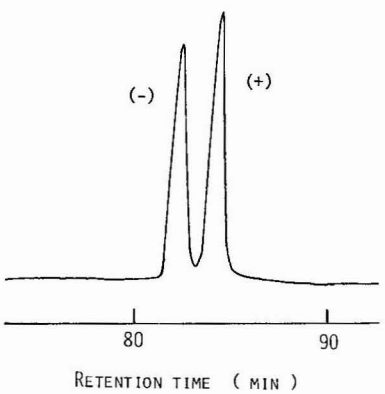



Fig. 1. Gas chromatogram of N-trifluoroacetyl-DL-leucine isopropyl ester. Glass capillary column (40 m × 0.25 mm I.D.) coated with di-*l*-menthyl (+)-tartrate. Temperature: 100°C. Carrier gas (helium) flow-rate: 0.7 ml/min.



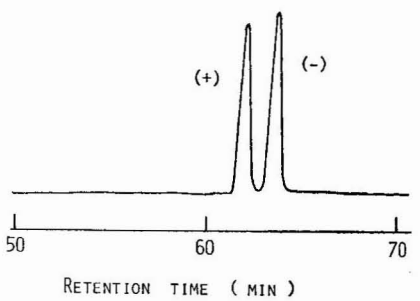




Fig. 2. Gas chromatogram of racemic N-pentafluoropropyl-α-phenylethylamine. Chromatographic conditions as in Fig. 1.

Fig. 3. Gas chromatogram of racemic α-bromo-β,β-dimethylbutyric acid *tert.*-butylamide. Chromatographic conditions as in Fig. 1.

groups linked to the asymmetric carbon atoms, as chiral stationary phases. This result supports the conclusion reported previously[4] that OH groups contribute to the formation of diastereomeric association complexes for the separation of enantiomers.

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## Note

# Determination of phenolic compounds in alternate fuel matrices

F. R. GUENTHER*, R. M. PARRIS, S. N. CHESLER and L. R. HILPERT
*National Bureau of Standards, Center for Analytical Chemistry, Washington, DC 20234 (U.S.A.)*
(Received October 17th, 1980)

The increased attention given alternate fuels, such as shale oil and solvent-refined coal, has given rise to the need for faster and more reliable methods of analysis for toxic compounds in these complex matrices. The high concentration of phenolic compounds found in these alternate fuels (relative to petroleum) has also increased the need for the development of reliable and rapid analytical procedures for these compounds.

The highly complex nature of the alternate fuel matrices requires high-resolution separation techniques to separate and identify the phenolic isomers present. Previous methods have involved the use of low-resolution packed columns[1–6], support-coated open-tubular (SCOT) columns[7], and wall-coated open-tubular (WCOT) columns[8–10], or the use of chemical derivatization and subsequent use of WCOT or packed columns[11–13].

In this paper we describe a method utilizing a high-resolution WCOT column for the separation and quantitation of phenols contained within a complex organic matrix. The utilization of this column, in combination with a simplified acid–base extraction scheme, produces a fast and reliable method for the quantitative analysis of phenols at $\mu$g/g (ppm) levels in alternate fuel matrices.

## EXPERIMENTAL*

Two alternate fuels, a shale oil and a solvent-refined coal (SRC) liquid, were characterized in this work. The shale oil was supplied by the Oakridge National Laboratory, Oakridge, TN, U.S.A., and is from the Mahogany zone of the Colorado Green River formation. It had been processed in a 150-ton retort for *in situ* simulated combustion operated by the Laramie Energy Research Center, Laramie, WY, U.S.A. The shale oil had undergone centrifugation to separate water (40%) and sludge before being received at our laboratory. The shale oil was then filtered, homogenized, and stored in amber ampoules.

The SRC sample is a middle-to-heavy distillate from a fuel oil blend obtained

* In order to specify procedures adequately, it has been necessary to identify some commercial materials in this report. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the material identified is necessarily the best available for the purpose.

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from the Pittsburg & Midway Coal Mining Co., Solvent-Refined Coal Pilot Plant, Dupont, WA, U.S.A. The SRC was similarly stored in amber ampoules for subsequent analysis.

*Extraction*

The alternate fuel sample (*ca.* 0.6 g) was accurately weighed into a small flask and dissolved in 50 ml of methylene chloride. An appropriate amount of *o*-chlorophenol, dissolved in methylene chloride, was then added to the sample as an internal standard. This solution was quantitatively transferred with an additional 50 ml of methylene chloride into a 250-ml separatory funnel. The acids were then isolated using an acid–base extraction procedure[14] adapted from Schmeltz[15]. Methylene chloride was substituted for ether in this procedure, because the stabilizing agent (2,6-di-*tert.*-butyl-*p*-cresol) in the ether interferred with final chromatographic quantitation. The resulting extract was dried over anhydrous sodium sulfate and concentrated under a stream of dry filtered nitrogen to 1 ml, in preparation for subsequent gas chromatographic (GC) analysis.

*Column preparation*

The analytical WCOT columns were prepared in our laboratory using the barium carbonate method of Grob *et al.*[16,17]. A 20 m × 0.3 mm I.D. capillary column was pulled from thick-walled borosilicate glass tubing. After the capillary had been acid-leached and dried, the inside surface was coated with $BaCO_3$ by forcing a saturated solution of barium hydroxide with $CO_2$ gas through the column. The column was then coated with a 20% solution of Pluronic L64 (Fluka, Buchs, Switzerland)[17] dissolved in methylene chloride. The column was conditioned at 220°C until it exhibited minimal bleed. Subsequent testing using the procedure described by Grob and Grob[18], revealed a film thickness of *ca.* 0.07 $\mu$m.

The gas chromatograph used for this work was equipped with a pressure-controlled capillary inlet system and a flame ionization detector. The chromatographic peaks were integrated using a digital integrator capable of internal standard calculations. The GC conditions used are listed in the caption of Fig. 1.

*Qualitative analysis*

Peak identification was accomplished utilizing a quadrupole GC–mass spectrometry (MS) system equipped with a 20 m × 0.3 mm I.D. Pluronic L64 WCOT column. GC conditions were identical with those used in quantitative analysis (see Fig. 1). The WCOT column was interfaced to the mass spectrometer through an "open-slit" fitting constructed out of nickel tubing (1/16 in. O.D. × 0.010 in. I.D.). The mass spectrometer was operated in the electron impact mode under the following conditions: electron energy, 70 eV; ionizing current, 1 mA; ion source manifold pressure, $1 \cdot 10^{-5}$ Torr; ion source temperature, 200°C; interface temperature, 250°C. Mass spectra were scanned repetitively every 2 sec under computer control for the entire GC run. Isomer identifications were verified by analysis of the mass spectra and comparisons of retention times of pure phenolic standards.

*Quantitative analysis*

Calibration factors of all the phenols relative to *o*-chlorophenol were deter-

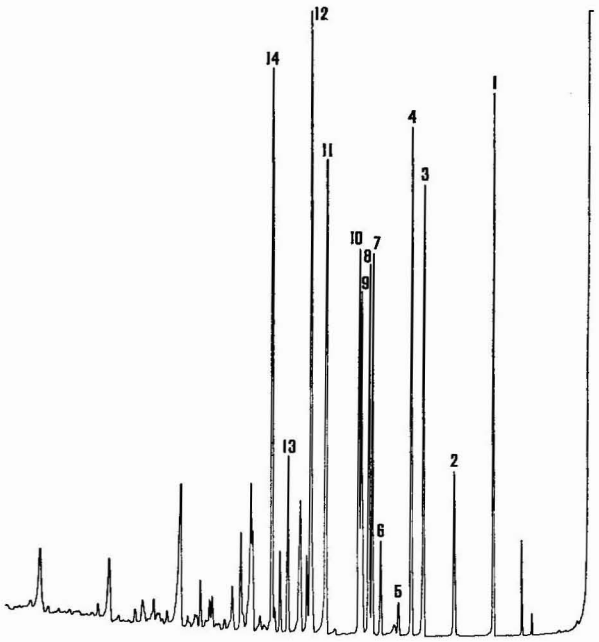


Fig. 1. Chromatogram of acidic fraction from shale oil. Column, 20 m × 0.3 mm I.D., Pluronic L64 WCOT; temperature program, 70°C to 160°C at 2°C/min; carrier gas, helium; split, 30:1; injector and detector temperatures, 300°C.

mined from a standard solution of gravimetrically prepared amounts of each of the analytes and *o*-chlorophenol in methylene chloride. Gold weighing boats were used in the preparation of standard solutions, after it was shown that using aluminium weighing boats introduced a large variability in the determination. The concentrations of the phenols in the standard solution were made to mimic the concentration of the phenols in the samples as closely as possible. An aliquot of this standard was extracted using the same procedure already described. Subsequent GC analyses yielded calibration factors which were then applied to the data from sample runs to yield quantitative results.

## RESULTS AND DISCUSSION

A chromatogram of the shale oil acids on the Pluronic L64 WCOT column is shown in Fig. 1. The chromatogram contains over 50 peaks, most of which are sufficiently resolved for peak area integration with a precision of 5% or better. Peaks 1–14 were identified by GC–MS and retention indices, and are listed in Table I. Of particular note is the separation of all six dimethylphenol isomers (xylenols), the cresols, and phenol. This separation was achieved using a temperature program of 70°C initial, programmed to 150°C at 2°C/min. All the $C_1$–$C_3$ phenols in the sample eluted prior to a column temperature of 130°C. Retention on this liquid phase seems to be heavily dependent on the amount of steric hindrance, by adjacent methyl groups, around the polar hydroxy group. This is demonstrated by the elution of the dimethyl-

TABLE I

IDENTIFICATION OF PHENOLIC COMPOUNDS IN FIG. 2, BOILING POINTS, EXTRACTION EFFICIENCIES AND ANALYTICAL RESULTS

n = Not determined.

| *Compound* | *Peak No. Fig. 1* | *B.p. (°C)* * | *Extraction efficiency* | *Concentration found (μg/g)* ** | |
|---|---|---|---|---|---|
| | | | | *Shale oil* | *SRC II fuel* |
| *o*-Chlorophenol | 1 | 176 | 99.1 | int. std. | int. std. |
| 2,6-Dimethylphenol | 2 | 212 | 74.9 | 168 ± 8 | 1450 ± 90 |
| Phenol | 3 | 182 | 99.3 | 401 ± 4 | 23,800 ± 1200 |
| *o*-Cresol | 4 | 191 | 98.1 | 384 ± 9 | 12,500 ± 500 |
| 2,4,6-Trimethylphenol | 5 | 219*** | n | n | n |
| 2,3,6-Trimethylphenol | 6 | — | n | n | n |
| *p*-Cresol | 7 | 202 | 99.3 | 273 ± 7 | 15,500 ± 800 |
| *m*-Cresol | 8 | 203 | 98.6 | 327 ± 10 | 29,100 ± 1900 |
| 2,5-Dimethylphenol | 9 | 242 | 93.4 | 320 ± 12 | 8900 ± 500 |
| 2,4-Dimethylphenol | 10 | 214 | 89.4 | 387 ± 17 | 8200 ± 600 |
| 2,3-Dimethylphenol | 11 | 218 | n | n | n |
| 3,5-Dimethylphenol | 12 | 219 | n | n | n |
| 3,4-Dimethylphenol | 13 | 225 | n | n | n |
| 2,3,5-Trimethylphenol | 14 | 233§ | n | n | n |

* Ref. 20.
** Uncertainties are ± 1σ.
*** Ref. 21.
§ Ref. 22.

substituted phenols. The sterically hindered 2,6-dimethylphenol (b.p., 212°C) elutes prior to phenol (b.p., 182°C), while the least hindered, 3,4-dimethylphenol (b.p., 225°C), is retained quite strongly.

The concentrations of several of the phenols in the shale oil and SRC are given in Table I. From this table it can be seen that the total phenolic contents of the shale oil and the SRC fuel oil are *ca.* 0.3 and 10%, respectively. The chromatogram of the SRC fuel oil is shown in Fig. 2. This chromatogram shows a very similar pattern of phenols to that of the shale oil. The individual concentrations, however, are approximately two orders of magnitude higher. These experimental results agreed with those determined by a method[14] using a liquid chromatographic pretreatment followed by GC–MS quantitation.

The extraction efficiencies were determined by the ratio of the peak areas of the residual phenols in the extracted organic solution of the standard phenols, with the phenols in the extract solution. The extraction efficiencies of these phenols are listed in Table I. All of the phenols showed an extraction efficiency in excess of 70%.

## CONCLUSIONS

The method described in this paper has been shown to be both a rapid and straightforward method for the analysis of phenolic compounds in complex matrices, such as shale oil. The WCOT column used was shown to be capable of yielding highly reproducible quantitative separations.

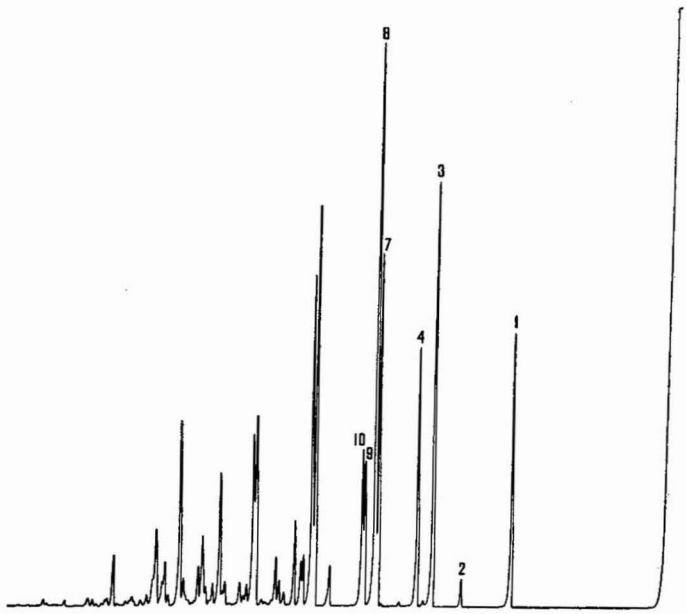


Fig. 2. Chromatogram of acidic fraction from a SRC II fuel oil. Same conditions as in Fig. 1.

It was experimentally determined that *ca.* 500 ng of phenol could be loaded onto the column without peak broadening or distortion. The large sample capacity and high resolving power exhibited by this column makes it ideal for GC–MS and GC Fourier transform infrared spectroscopic applications.

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## Note

# Use of a flame thermionic detector in the determination of glucosamine and galactosamine in glycoconjugates by gas chromatography

TOSHIAKI SHINOHARA
*National Research Institute of Police Science, Sanban-cho, Chiyoda-ku, Tokyo (Japan)*
(Received October 6th, 1980)

Gas chromatography (GC) is a convenient tool for the elucidation of carbohydrate constituents of glycoproteins or glycolipids. Recently, trifluoroacetic anhydride (TFAA) has been used as an acylating reagent in the GC separation and determination of alditols[1,2]. Acylated alditols have also been determined on a XF-1105 column in GC of human gastric mucopolysaccharides[3]. Tamura *et al.*[4] have developed this acylation method for an analysis of amino sugars. However, it seems likely that no satisfactory result has been obtained in the determination of amino sugars from natural origins, probably because of the instability to the trifluoroacetyl derivatives.

This paper describes a rapid determination method for glucosamine and galactosamine using a nitrogen-specific flame thermionic detector (FTD), and gives appropriate GC conditions for the simultaneous determination of the neutral and amino sugars in glycoconjugates of human origins.

## EXPERIMENTAL

### *Materials*

Standard neutral and amino sugars used were all commercial products: group A, L-fucose, L-arabinose, D-mannose, D-galactose, D-glucosamine hydrochloride, D-galactosamine hydrochloride; group B, L-rhamnose, D-xylose, D-glucose, D-mannosamine hydrochloride. The following biological materials were also investigated: crystalline ovomucoid (trypsin inhibitor from chicken egg white; Sigma, St. Louis, MO, U.S.A.); urinary crude glycopeptides[5] obtained from blood group O-secretor (U-SY), A-secretor (U-MS) and B-secretor (U-TS); partially purified glycoproteins[6] of blood group H-active (No. 5, Fr. I), A-active (No. 3, Fr. III) and B-active (No. 2, Fr. I) substances isolated from human ovarian cyst fluids according to the phenol extraction method[7]. *p*-Aminophenol hydrochloride was used as an internal standard.

A Shimadzu GC-7A gas chromatograph with FTD or a flame-ionization detector (FID) was used. The glass chromatographic columns were as follows: a, 2 m × 3 mm I.D., packed with 5% OV-101 on Chromosorb W AW DMCS (60–80 mesh); b, 1.5 m × 3 mm I.D., with 2% XF-1105 on Gas-Chrom P (60–80 mesh); c, 2 m × 3 mm I.D., with 2% QF-1 on Gas-Chrom P (60–80 mesh), modified by the chromatographic system[4].

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*Methods*

*Preparation of derivatized sample solution for GC.* To 1 ml of an aqueous solution containing 30–200 $\mu$g of each of the neutral and amino sugars (group A) was added 1 ml of 2% $NaBH_4$ containing 0.025 *M* NaOH in water, and the mixture was allowed to stand for 2 h at room temperature. After reaction, the excess of $NaBH_4$ was destroyed by adding 0.5 *N* HCl and the solution was concentrated, with several additions of methanol to remove methyl borate. The residue was dissolved in a small amount of water and the solution was applied to a column (8 × 1 cm I.D.) of QAE-Sephadex gels (borate form)[8], followed by elution with 25–30 ml of water. The eluent was discarded. The adsorbed alditols and amino alcohols on the gels were then eluted with concentrated HCl–methanol (1:24 v/v) until the eluent was completely replaced by this mixture, monitoring with a pH-test paper. The eluent was pooled and concentrated on an evaporator to yield a sugar alcohol fraction containing a considerable amount of methyl borate.

The concentrated residue was again dissolved in a small amount of methanol, followed by evaporation with several additions of the solvent to remove the borate. To the residue were added 0.2 ml of *p*-aminophenol in ethanol solution (360 $\mu$g/ml), and the mixture was completely transferred to a small capped glass tube (5 cm × 5 mm I.D.) by repeated washings with a small amount of methanol and water. The mixed solution was concentrated to dryness by evaporation and standing in a vacuum desiccator over $P_2O_5$. The dried matter was suspended in 50 $\mu$l of ethyl acetate, and 50 $\mu$l of TFAA were added with cooling on ice. The mixture was allowed to stand at room temperature for 30 min, and an aliquot of the reacted solution (1–2 $\mu$l) was applied to the chromatographic column.

The standard sugar mixture of group B was similarly treated and the prepared trifluoroacetate derivatives were analyzed by GC.

*GC conditions.* Standard calibration graphs for the hexosamine assays were made by using the OV-101 column under the following operating conditions in the FTD system: injector and detector temperature, 210°C; column temperature, 120°C (isothermal); nitrogen carrier gas flow-rate, 50 ml/min; hydrogen flow-rate, 6–8 ml/min; air flow-rate, 220 ml/min; electrical heating on an alkali-metal salt bed. A conventional FID system was also employed. The calibration graphs for the determination of the neutral sugars were obtained on the XF-1105 column with temperature programming from 100°C to 160°C at 2°C/min in the FID system under the operating conditions indicated in Table I. The internal standard (*p*-aminophenol hydrochloride) was used for both calibrations. Relative retention times ($R_t$, min) of the neutral or amino sugars on the chromatographic columns a–c were recorded with respect to the standard compound (Table I).

*Determination of carbohydrate constituents of glycoconjugates.* A 1–2 mg amount of each of the urinary crude glycopeptides, human ovarian cyst glycoproteins and ovomucoid were hydrolyzed in 4 *N* trifluoroacetic acid (TFA) for 16 h at 100°C in sealed tubes. After hydrolysis, TFA was removed by evaporation with repeated additions of water. The hydrolyzates were each treated with the reducing reagent, and the resultant alditols and amino sugar alcohols were subjected to clean-up on the QAE-Sephadex column, as described for the standard sugar alcohols. To the final methanol–HCl eluent was added an ethanol solution of *p*-aminophenol (36–180 $\mu$g). The eluent was evaporated with repeated additions of methanol to remove methyl

TABLE I

RELATIVE RETENTION TIMES IN SEPARATIONS OF SUGAR MIXTURES

Gas chromatograhhic conditions as in the text.

| *Sugar* | *Column a, OV-101 120°C* | *Column b, XF-1105* | | *Column c, QF-1 170°C* |
|---|---|---|---|---|
| | | *100–160°C* | *170°C* | |
| Rhamnose | —* | 0.60 | —* | —* |
| Fucose | 0.27 | 0.67 | — | 0.56 |
| Arabinose | 0.25 | 0.87 | — | 0.65 |
| Xylose | — | 0.95 | — | — |
| Mannose | 0.34 | 1.12 | — | 0.91 |
| Glucose | — | 1.27 | — | — |
| Galactose | 0.37 | 1.32 | — | 1.06 |
| Glucosamine | 0.63 | dec.** | 4.12 | 2.29 |
| Galactosamine | 0.71 | dec. | 4.71 | 2.62 |
| Mannosamine | 0.69 | dec. | 5.29 | 2.33 |
| *p*-Aminophenol (min) | 1.00 (11.5) | 1.00 (16.2) | 1.00 (1.7) | 1.00 (3.4) |

* Not examined.

** Considerably decomposed during the development.

borate. The residue was transferred to a small glass tube, followed by acylation with TFAA as described above. A portion of the resulting solution (1–2 $\mu$l) was used for chromatography. Determinations of the hexosamine contents of these biological samples were carried out on the OV-101 column (a) at 120°C in the FTD system. Determination of the content of neutral sugars were performed on the XF-1105 column (b) at 100–160°C (2°C/min) in the FID system.

## RESULTS AND DISCUSSION

Fig. 1 shows the calibration graphs for glucosamine and galactosamine obtained by using FTD or FID. From these the detection limits for glucosamine or galactosamine were *ca.* 20 ng with the FTD system and *ca.* 100 ng with the FID. The sensitivity of FTD for the amino compounds may generally be varied by changing the detector arrangement; thus it is possible to raise the detector response at least ten times higher than the FID response by electrically heating the bed of an alkali source.

Table I gives the $R_t$ values of the neutral and amino sugars on these chromatographic columns, with respect to *p*-aminophenol. The neutral monosaccharides were satisfactorily separated and determined on the XF-1105 column with temperature programming from 100°C to 160°C, using the FID system. However, amino sugars were considerably decomposed during the development under this conditions. There was little decomposition or adsorption of the amino sugars on this column in a rapid analysis and good separations were obtained at 170°C, with only slightly inferior reproducibility. Determination of glucosamine and galactosamine was satisfactorily performed with column a packed with the non-polar stationary phase OV-101 without degradation or adsorption on the column at the lower temperature (120°C). However, in the simultaneous separation of glucosamine, mannosamine, and galactosamine the $R_t$ values obtained were 0.63, 0.69 and 0.71, respectively.

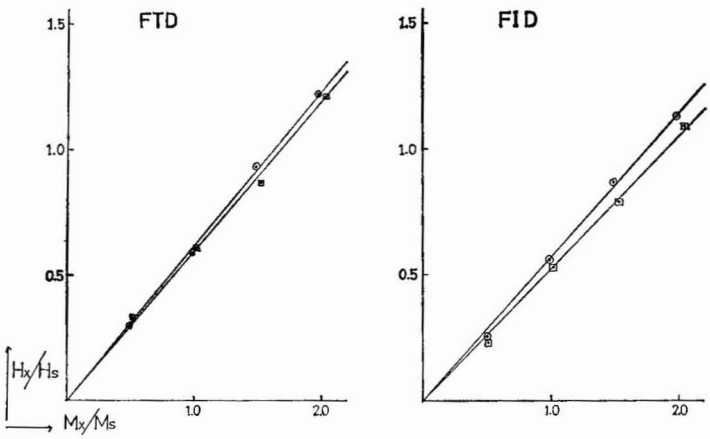


Fig. 1. Calibration graphs for determination of glucosamine (○) and galactosamine (□). Internal standard: *p*-aminophenol hydrochloride (72 $\mu$g). $H_x/H_s$, $M_x/M_s$ = Peak height ratio and weight ratio of hexosamine to internal standard. Calibration was performed with a chromatographic column (2 m × 2 mm I.D.) packed with 5% OV-101 on Gas-Chrom P at 120°C. with either FTD (left graph) or FID (right graph).

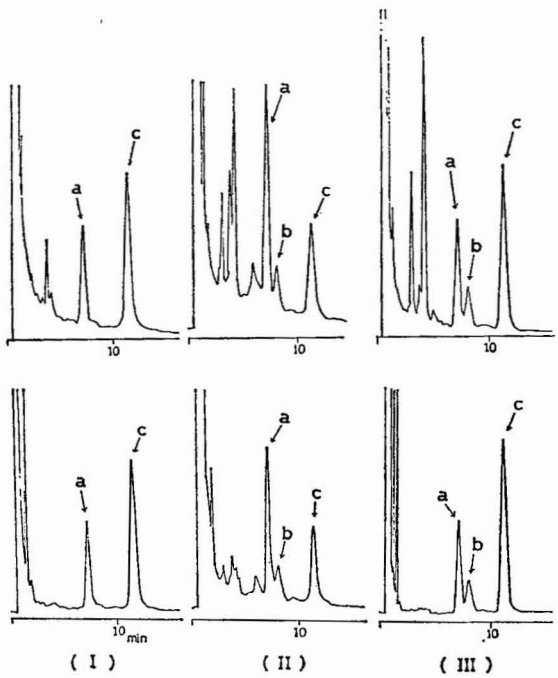


Fig. 2. Comparison of detector response to the hexosamine assays for ovomucoid (I), urinary glycopeptides (U-SY) (II) and ovarian cyst glycoprotein (No. 2, Fr. I) (III). Upper chromatograms: FID system. Lower chromatograms; FTD system. Analyses were accomplished with the OV-101 column at 120°C, and the data are indicated in Table II. Peaks: a = glucosamine; b = galactosamine; c = internal standard (*p*-aminophenol; I, II, 72 $\mu$g; III, 180 $\mu$g).

Fig. 2 shows the chromatographic profiles of ovomucoid (I), crude urinary fraction (U-SY) (II) and ovarian cyst glycoprotein (No. 2, Fr-I) (III) for comparison at the FID and FTD systems. The upper chromatogram was run with the FID and the lower with the FTD. In the FTD mode, glucosamine (a) and galactosamine (b) were selectively detected and well determined with the internal standard method. The standard *p*-aminophenol (c) was found to be suitable, having good stability and appropriate $R_t$ value, for the determination of both amino sugars. Table II indicates the carbohydrate composition of the experimental biological materials. The monosaccharide composition of ovomucoid was estimated as about half that of the purified ovomucoid as reported in the literature[9]. This may be due to the heterogeneity of the mucoid or the difference in the hydrolysis conditions.

TABLE II

CARBOHYDRATE CONTENTS (%) IN GLYCOCONJUGATES

U.G. = Urinary crude glycopeptides; O.G. = ovarian cyst glycoproteins. The origins of the materials and the experimental conditions are as in the text. A minus sign indicates a value below the detection limit.

| *Material* | *Fucose* | *Mannose* | *Galactose* | *Glucosamine (as hydrochloride)* | *Galactosamine (as hydrochloride)* |
|---|---|---|---|---|---|
| Ovomucoid | — | 2.7 | 0.7 | 8.6 | — |
| U.G. (U-SY) | 1.2 | 1.9 | 3.1 | 7.0 | 1.5 |
| U.G. (U-MS) | 0.6 | 1.6 | 1.8 | 6.3 | 2.0 |
| U.G. (U-TS) | 0.6 | 1.0 | 2.1 | 4.2 | 0.9 |
| O.G. (No. 5, Fr. I) | 14.0 | 1.0 | 14.4 | 18.0 | 3.4 |
| O.G. (No. 3, Fr. III) | 12.0 | — | 24.6 | 30.1 | 7.7 |
| O.G. (No. 2, Fr. I) | 3.5 | 1.0 | 9.5 | 7.7 | 2.8 |

Acylation of neutral and amino sugar alcohols is often performed with acetic anhydride, as for the chemical characterization of glycoprotein[10] or glycolipid[11]. Acetylated amino alcohols are chemically more stable than the trifluoroacetylated compounds. However, they may lead to problems such as time-consuming preparation or column temperatures over 220°C in GC. Nevertheless, the trifluoroacetates of glucosamine and galactosamine were satisfactorily analyzed at the lower column temperature without any degradation. There are a few problems in the separation and determination of the amino sugars in glycoconjugates in this experimental system: (1) reduction to the amino alcohols requires the somewhat rigorous treatment with 2% $NaBH_4$ in 0.025 *M* NaOH; (2) impurities must be completely removed from the amino alcohols with QAE-Sephadex gel (borate form) filtration; (3) a suitable chromatographic column packed with a non-polar stationary phase such as the silicone OV-101 is required for a satisfactory determination.

The usefulness of FTD, already acknowledged in the determination of nitrogen- and phosphorus-containing compounds[12,13], has again been demonstrated in this hexosamine assay.

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## Note

# Separation of neutral and amino sugars by capillary gas chromatography

C. GREEN and V. M. DOCTOR*
*Department of Chemistry, Prairie View A&M University, Prairie View, TX 77445 (U.S.A.)*
G. HOLZER
*Department of Chemistry and Geochemistry, Colorado School of Mines, Golden, CO 80401 (U.S.A.)*
and
J. ORÓ
*Departments of Biophysical Sciences and Chemistry, University of Houston, Houston, TX 77004 (U.S.A.)*
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Analysis of neutral sugars of the glycoproteins by gas chromatography (GC) and of the amino sugars from the same sample by ion-exchange chromatography was the most widely accepted procedure[1] until Niedermeir[2] first separated the hexosamines in glycoproteins by GC of the alditol acetate derivatives. Although glucosamine was separated from galactosamine by this method with good resolution, mannosamine was not separated from galactosamine. Also, rhamnose was not resolved from fucose and ribose was not resolved from arabinose. These workers used a 6 × ¼ in. glass column packed with 1% ECNSS-M on Gas-Chrom A. Griggs *et al.*[3] reported identical results using Gas-Chrom P (100–200 mesh) precoated with a mixture of 0.2% ethylene glycolsuccinate, 0.2% stabilized ethylene glycol adipate and 1.4% silicone XE-60 with temperature programming, beginning at 150°C with a program rate of 1°C/min to a final temperature of 205°C. A separation of 6 neutral and 2 amino sugars was reported by Metz *et al.*[4] who used OV-225 and determined the optimum hydrolysis conditions for their recovery from glycoprotein samples. Niedermeir and Tomana[5] in a subsequent study reported an effective separation of the alditol acetates of the three hexosamines using a polyamide (Poly A 103) liquid phase, but galactose was not separated from glucose and rhamnose and ribose were not included in the known mixture. A common constituent of acidic polysaccharides of plant gums and hemicelluloses has been identified as 4-O-methyl-D-glucuronic acid. One of the procedures[6] for its determination involved reduction of the polysaccharide before hydrolysis and the 4-O-methyl glucose thus formed was subsequently identified as its alditol acetate derivative. Holzer *et al.*[7], using a 20 m × 0.3 mm glass capillary column coated with a 9:1 mixture of N-propionyl-L-valine-*tert.*-butylamide polysiloxane and Witoconol LA23, with temperature programming from 90–200°C at 6°C/min, reported a separation of alditol acetates of the common neutral sugars. However, when this procedure was applied to the analysis of plant gums and hemicelluloses which contain 4-O-methyl-D-glucuronic acid, the alditol acetates of mannose and 4-O-methyl-D-glucose were not fully separated[7].

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In the present report a chiral stationary phase was used for the first time during gas–liquid chromatographic (GLC) analysis of a mixture of neutral sugars and the three hexosamines. By using a longer glass capillary column of 35 m × 0.3 mm coated with the same mixture, a complete separation of the alditol acetates of the common neutral sugars including 3-O-methyl- and 4-O-methyl-D-glucitols was accomplished. An application of this method for the separation of the alditol acetates from the hydrolysate of reduced polysaccharide from *Daemonorops* species is described.

## MATERIALS AND METHODS

GC was carried out on a Varian Aerograph 2000, adapted for glass capillary work. In addition, the alditol acetates were analyzed by GLC–mass spectrometry (MS) using an LKB instrument. Identification was done by a comparison of retention time data and mass spectral fragmentation patterns with those of known standard. The glass capillaries were drawn from Pyrex glass, having a I.D. of 0.3 mm. For the analysis of the alditol acetates, columns of 20–35 m were etched with 5% $KHF_3$ solution[8] and deactivated using the Carbowax 20M method[9]. The column was then coated with a 0.2% stationary phase, consisting of 90% N-propionyl-L-valine-*tert.*-butylamide polysiloxane and 10% Witconal LA 23 as surfactant using the static method. The column was conditioned at 230°C with a low helium carrier gas flow-rate. For the analysis of the alditol acetates the column was operated at helium flow-rates between 4 and 6 ml/min and temperatures up to 200°C.

Solutions (0.05 *M*) of alditol acetates of neutral sugars were purchased from Regis (Morton Grove, IL, U.S.A.) while the remaining monosaccharides were derivatized by the procedure outlined in the *Operation Manual* supplied by the above company. Polysaccharide material (B-fraction) was isolated from benzene extracted hemicellulose of *Daemonorops* species after releasing it during the delignification by the method of Whistler *et al.*[10]. The purified polysaccharides were reduced by reaction of the propionated methyl ester with lithium borohydride in tetrahydrofuran, hydrolyzed with sulfuric acid, reduced with sodium borohydride and acetylated by the procedure of Dutton and Kabir[6].

## RESULTS AND DISCUSSION

Chiral polysiloxane phases were introduced by Frank *et al.*[11,12] for the separation of enantiomeric amino acids. The polarity of the phase and its thermal stability make it useful for the analysis of a variety of compounds[13].

Gas chromatograms of the mixture of alditol acetates of the 13 common sugars and the three hexosamines is shown in Fig. 1. The peaks were identified by co-chromatography and GLC–MS. The results showed that except for the acetates of D-mannitol and 4-O-methyl-D-glucitol, all of the other alditol acetates were well separated by this method. The procedure can be applied for the identification of sugars in the glycoproteins which contain one or more neutral and amino sugars.

In order to obtain a complete separation of D-mannitol and 4-O-methyl-D-glucitol, the sample mixture of 13 alditol acetates was injected in a 35 m × 0.3 mm glass capillary column. The results presented in Fig. 2 showed that all of the components were fully separated. The longer column used in this study prolonged the

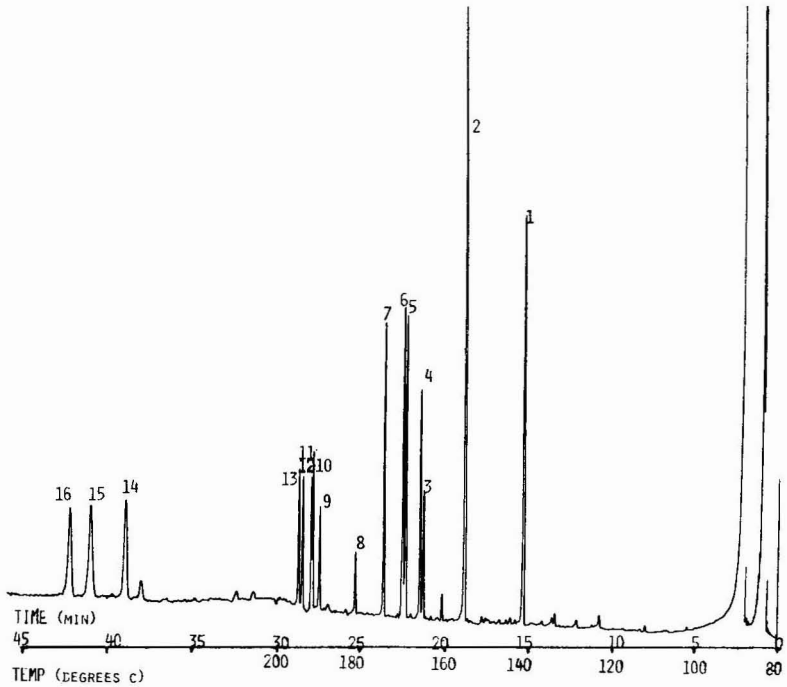


Fig. 1. Gas chromatograph of alditol acetates of the neutral sugars and the three hexosamines. Column: 20 m × 0.3 mm glass capillary column coated with 9:1 mixture of N-propionyl-L-valine-*tert*-butylamide polysiloxane and Witconal LA 23. Temperature: 80–200°C at 4°C/min for 30 min and isothermal at 200°C. Helium pressure: 18 p.s.i., flame-ionization detector. Peaks: 1 = erythritol; 2 = D-2-deoxyribitol; 3 = L-rhamnitol; 4 = L-fucitol; 5 = ribitol; 6 = arabitol; 7 = xylitol; 8 = D-2-deoxyglucitol; 9 = 3-O-methyl-D-glucitol; 10 = 4-O-methyl-D-glucitol; 11 = D-mannitol; 12 = D-galactitol; 13 = D-glucitol; 14 = N-acetyl glucitol; 15 = N-acetyl-galactitol; 16 = acetyl mannitol.

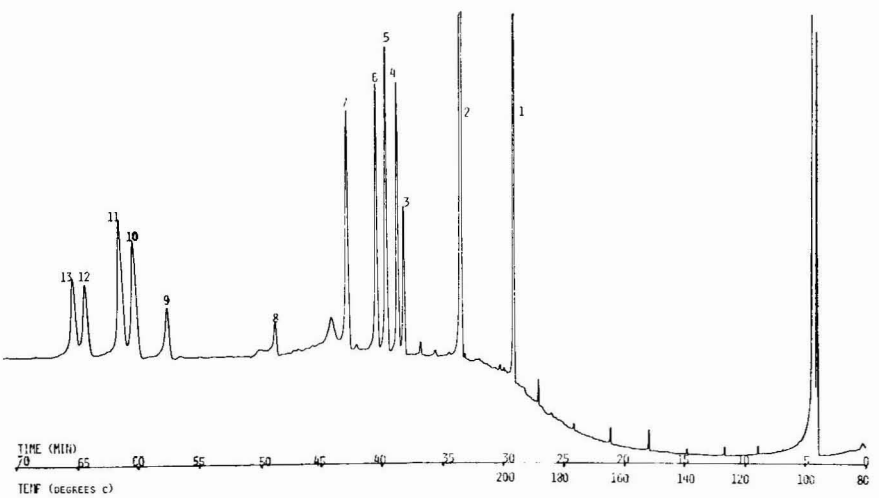


Fig. 2. Gas chromatograph of alditol acetates of the neutral sugars on a 35 m × 0.3 mm glass capillary coated with the same mixture as in Fig. 1. Temperature program: 80–200°C at 4°C/min and isothermal at 200°C; Peaks 1–13 same as in Fig. 1.

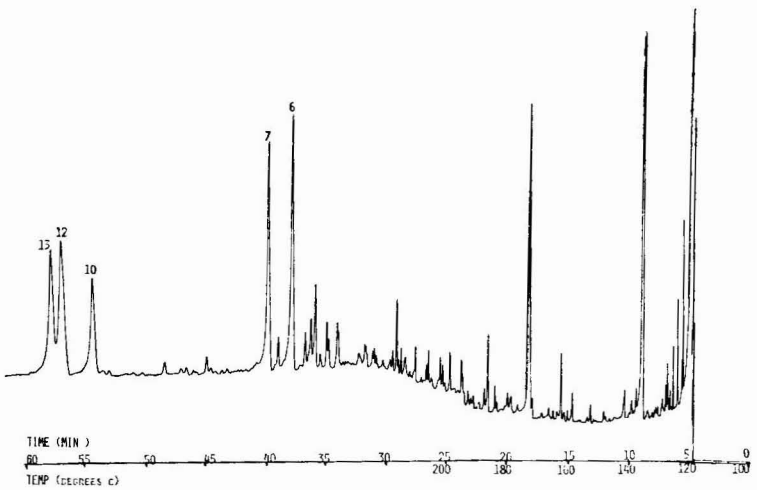


Fig. 3. Gas chromatograph of alditol acetates from the hydrolysate of reduced polysaccharide from *Daemnorops* species. Column, helium pressure and peaks were the same as in Fig. 2. Temperature program: 100–200°C at 4°C/min and isothermal at 200°C.

analysis time in comparison with Fig. 1 but a complete separation of all sugars was obtained. This procedure would be recommended for the analysis of sugars in plant gums or hemicelluloses which may contain 4-O-methyl-D-glucuronic acid and one or more neutral sugars.

The GLC profile of the alditol acetates of the sugars from the hemicellulose of *Daemonorops* species reported in Fig. 3 showed the presence of arabinose, xylose, 4-O-methyl-D-glucuronic acid, galactose and glucose. The results reported[7] earlier with the 20-m capillary column showed that D-mannitol and 4-O-methyl-D-glucitol were so close to each other that without mass spectral fragmentation pattern, the two could not be identified. In this study, however, by GLC alone or by co-chromatography, the two components could be easily identified. The components which emerged between 120 and 180°C before the alditol acetates may be the result of impurities in the polysaccharide or from the reagents used in the isolation of the sugars. The advantage of the chiral phase in the analysis of alditol acetates is its potential thermal stability as well as good resolving power.

ACKNOWLEDGEMENTS

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## Note

# Capillary gas chromatographic separation of monosaccharides as their alditol acetates

J. KLOK*, E. H. NIEBERG-VAN VELZEN, J. W. DE LEEUW and P. A. SCHENCK
*Delft University of Technology, Department of Chemistry and Chemical Engineering, Organic Geochemistry Unit, De Vries van Heystplantsoen 2, 2628 RZ Delft (The Netherlands)*
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During organic geochemical research dealing with the occurrence and composition of polysaccharides in recent marine sediments, a gas–liquid chromatographic (GLC) separation is required by which mixtures of monosaccharide derivatives can be baseline separated.

To avoid complex mixtures of anomeric monosaccharides, the hydrolytically released monosaccharides can be reduced to the corresponding alditols. The alditol mixture is subsequently derivatized into the alditol acetates. The GLC separation of alditol acetates on columns packed with OV-275[1] and ECNSS-M[2–5] has been reported. In the latter case the column is usually operated under conditions close to the maximum operating temperature, which limits column life[6]. Moreover, a baseline separation of some of the alditols used in these studies is not achieved. Holzer *et al.*[7] applied a glass capillary column coated with a chiral phase. Their results show that the separation of rhamnitol/fucitol and ribitol/arabitol is not complete. This report describes the baseline separation of ten alditol acetates using a glass capillary column coated with OV-275.

## EXPERIMENTAL

The ten alditols used as standards in this study are listed in Table I. They are commercially available from various companies. The standard mixture of the alditol acetates was prepared by acetylation of a mixture of the alditols, containing equal amounts (by weight) of the individual alditols. The acetylation was performed in a

TABLE I

THE ALDITOLS USED AS STANDARDS IN THIS STUDY

| *Alditol* | *No.* | *Alditol* | *No.* |
|---|---|---|---|
| Erythritol | 1 | Xylitol | 6 |
| Rhamnitol | 2 | Mannitol | 7 |
| Fucitol | 3 | Galactitol | 8 |
| Ribitol | 4 | Sorbitol | 9 |
| Arabitol | 5 | *m*-Inositol | 10 |

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sealed vial with pyridine–acetic anhydride (1:1) at 100°C during 2 h. After evaporation of the acetylation reagent the alditol acetate mixture was dissolved in dichloromethane.

The natural mixture of monosaccharides was obtained from a diatomaceous ooze sample from the Namibian Shelf (S.W. Africa, 22°51.5′ S, 14°14.5′ E)[8]. The sample was lyophilized and hydrolysed with 1 *M* $H_2SO_4$ during 3 h at 100°C. The hydrolysate was neutralized with $BaCO_3$ and reduced with $NaBH_4$. Subsequent acetylation was performed as described above.

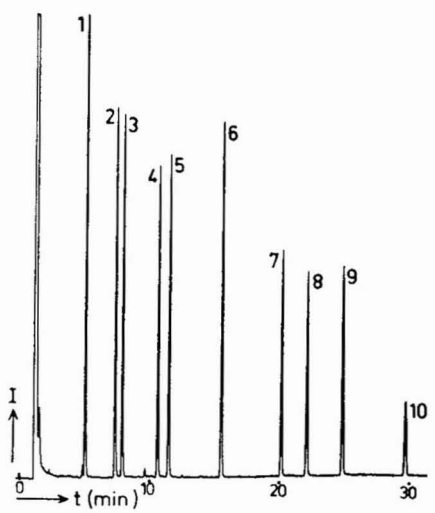
GLC was carried out on a Varian 3700 gas chromatograph equipped with a glass capillary column (25 m × 0.25 mm I.D.) coated with OV-275 (Chrompack, Middelburg, The Netherlands). The temperature was programmed from 190 to 215°C at 1°C/min. Further GLC conditions: injector, 250°C; flame ionization detector, 250°C; carrier gas, helium at a flow-rate *ca.* 1.5 ml/min; helium pressure, 18 p.s.i.; splitter, 30 ml/min; attenuator, $1 \cdot 10^{-11}$ mA.

Identification of the acetates was based on the retention times of the individual alditol acetates.

## RESULTS AND DISCUSSION

Fig. 1 shows the gas chromatogram of the standard alditol acetate mixture. The peak numbers correspond to the alditols listed in Table I. All components are baseline separated, thus allowing a complete qualitative and quantitative analysis of monosaccharides as their alditol acetates.

Fig. 2 shows the gas chromatogram of the mixture obtained from the diatomaceous ooze sediment after hydrolysis, reduction and derivatization. *m*-Inositol was added as an internal standard. The relative retention times of the main peaks corre-



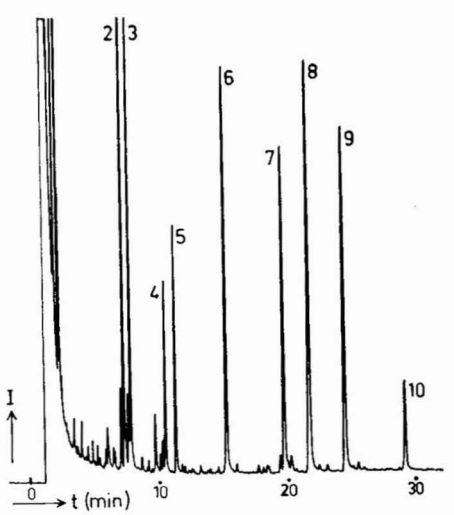




Fig. 1. Gas chromatogram of a standard mixture of ten alditol acetates. The peak numbers correspond to the alditols listed in Table I.

Fig. 2. Gas chromatogram of the alditol acetates obtained from a recent sediment. *m*-Inositol (10) was added as an internal standard. The peak numbers correspond to the alditols listed in Table I.

spond exactly to those of the alditol acetates in Fig. 1. Ultimate identification of both major and minor peaks has to be achieved by GLC–mass spectrometry.

The abundance of rhamnose and fucose in the ooze sample is not unexpected since these monosaccharides are major building blocks of algal polysaccharides[9–12].

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## Note

# Separation of proteins on silicone-coated porous glass

TAKAHARU MIZUTANI
*Faculty of Pharmaceutical Sciences, Nagoya City University, Mizuho-ku, Nagoya 467 (Japan)*
(Received October 27th, 1980)

Porous glass, which was developed for exclusion chromatography[1], adsorbs proteins and is used for adsorption chromatography of proteins[2]. To prevent such adsorption, porous glass can be siliconized and used for exclusion chromatography of proteins[3]. The coated glass adsorbs hardly any protein at low concentrations of salts. However, at high concentrations of salts, the coated glass adsorbs significant amounts of protein by hydrophobic bonding[4]. Lymphocytes can be separated on siliconized glass beads[5,6]. This paper reports the separation of proteins on siliconized porous glass.

## MATERIALS AND METHODS

The porous glass used was CPG-10 240 Å (Electro-Nucleonics, Fairfield, NJ, U.S.A.). After being washed with a chromic acid mixture and then water, the glass was dried at 180°C. Then 1 g of dried glass was mixed with 3 ml of carbon tetrachloride containing 50 mg of silicone oil (dimethylpolysiloxane, KF 96; Shi-Etsu Chemicals, Tokyo, Japan). After evaporation of the carbon tetrachloride, the glass was tightly coated with silicone by heating at 300°C for 5 min. The surface area of coated glass was measured to be 51.7 $m^2/g$ of the glass, using an Orr Surface-Area Pore-Volume Analyzer (Micromeritics, Norcross, GA, U.S.A.) with nitrogen gas.

The coated glass was precipitated in 1 % sodium dodecyl sulphate (SDS)–0.2 *M* phosphate solution (pH 7.4) and the precipitated glass was packed in a column (4.5 × 0.75 cm I.D.; 1 g, 2 ml)[3]. The SDS was removed by thoroughly washing with *ca.* 100 column volumes of degassed hot water[7].

A solution of 1 ml containing 5 mg of hemoglobin (Sigma, St. Louis, MO, U.S.A.) and 5 mg of bovine serum albumin (Miles, Elkhart, IN, U.S.A.) was applied to the column, which was previously equilibrated with buffers. After the elution of the buffer, the protein adsorbed was eluted with 1 % SDS–0.2 *M* phosphate at pH 7.4. The fraction volume collected was 1 ml and the elution was carried out at room temperature. The recoveries of total protein and albumin were determined by the measurements of the absorbance at 280 nm. The recovery of hemoglobin was determined from the absorbance at 541 nm. Globulin was prepared from bovine serum by fractional precipitation with ammonium sulfate (20 %, w/w). Globulin (10 mg) was dissolved in 2 ml of 0.01 *M* phosphate (pH 7.4) and the solution was applied to the column. Proteins in the fractions were analyzed by polyacrylamide gel disc electro-

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phoresis for albumin and hemoglobin[8] or by electrophoresis on cellulose acetate for globulin. The stained gels and cellulose acetates were treated with an autodensitometer (Fujiriken).

## RESULTS AND DISCUSSION

Fig. 1 shows the elution patterns of a mixture of bovine hemoglobin and albumin on the silicone-coated porous glass columns. The proteins were only slightly eluted with 10 m*M* NaCl and eluted partially with 1% SDS–0.2 *M* phosphate solution (Fig. 1A). The overall recovery of proteins was 34% and that of hemoglobin was 16%, from the measurement of the absorbance at 541 nm.

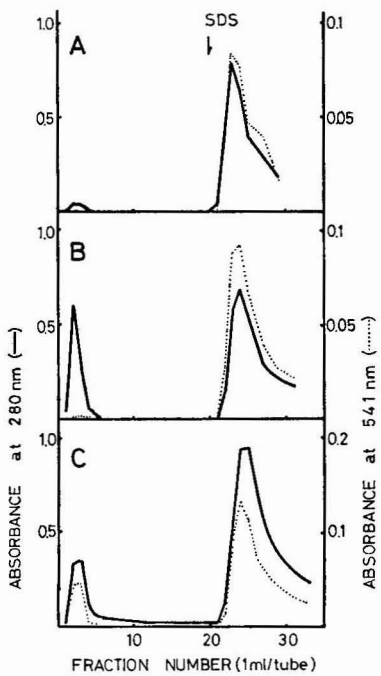


Fig. 1. Elution profiles of a mixture of albumin (5 mg) and hemoglobin (5 mg) on a silicone-coated porous glass column (4.5 × 0.75 cm I.D.). The buffers used were (A) 10 m*M* NaCl–0.2 m*M* phosphate (pH 7.4); (B) 5 m*M* Tris–HCl (pH 7.6); (C) 5 m*M* Tris–HCl (pH 7.6) containing 10 m*M* glutamic acid. At fraction 20 of each chromatogram, the columns were eluted with 1% SDS–0.2 *M* phosphate (pH 7.4).

Fractions 2 and 3 in Fig. 1B, eluted with 5 m*M* Tris–HCl, did not contain hemoglobin since the fractions did not show the absorbance at 541 nm of hemoglobin. Fig. 2 shows the result of tracing of the disc gel stained of fraction 2 in Fig. 1B, and the pattern indicates that fraction 2 does not contain hemoglobin but only albumin. The recovery of albumin in fractions 2 and 3 in Fig. 1B was 32%. More proteins loaded on the column were eluted with 1% SDS–0.2 *M* phosphate. The overall recovery of proteins was 42% and that of hemoglobin was 19%.

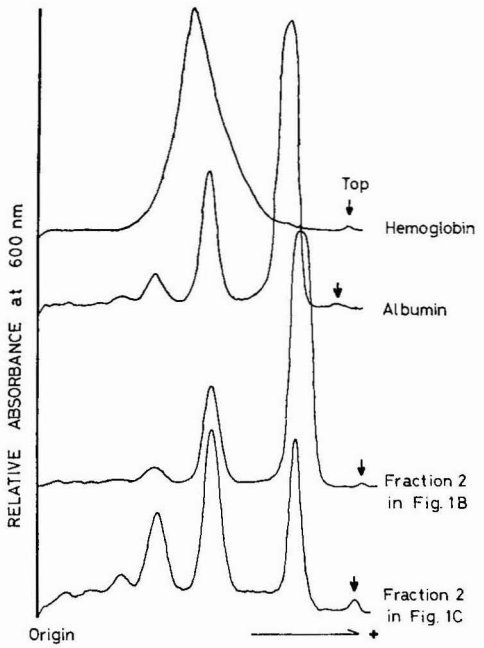


Fig. 2. Densitometric tracing of stained disc gels of proteins in Fig. 1.

Fig. 1C shows the results of the elution with the Tris–HCl buffer containing 10 m*M* glutamic acid, to prevent adsorption. Some of the hemoglobin passed through the column, as indicated by the adsorbance at 541 nm. As shown in Fig. 2, the disc gel pattern of fraction 2 in Fig. 1C indicated the contamination of hemoglobin with albumin. However, more proteins were eluted with the SDS solution. The overall recovery of proteins was 61% and that of hemoglobin was 31%. The results in Fig. 1C indicate non-separation of proteins, even though the recovery was better.

Fig. 3 shows the elution patterns of bovine globulin in 0.01 *M* phosphate on silicone-coated glass. The electrophoretic patterns on cellulose acetate of fractions 2–5 in Fig. 3 is shown in Fig. 4, with the results of bovine serum and raw materials of globulin in Fig. 3. The recovery of proteins in fractions 2–5 was 25% of the proteins loaded on the column. The electrophoretic patterns in Fig. 4 indicate that the main

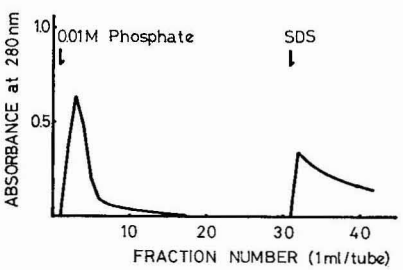


Fig. 3. An elution profile of globulin (10 mg) on a silicone-coated porous glass column (4.5 × 0.75 cm I.D.) in 0.01 *M* phosphate (pH 7.4).

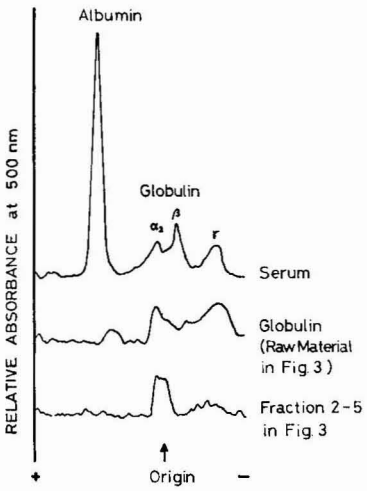


Fig. 4. Densitometric tracing of stained cellulose acetate of proteins in Fig. 3.

globulin in fractions 2–5 was $\alpha_2$-globulin. The $\beta$- and $\gamma$-globulins were not eluted with phosphate buffer from the column, and some parts were not eluted with the SDS solution since $\gamma$-globulin has a strong affinity for silicone-coated glass. Also, the Fc portion of surface immunoglobulin binds to detergent by hydrophobic bonding[9].

These results show that some proteins are separable on silicone-coated porous glass after careful selection of buffer. However, the method does not give complete elution of proteins adsorbed on the glass with natural buffers. The system effectively separates proteins of low affinity from proteins of high affinity. In these experiments, 1 g of glass was used for the separation of 10 mg of a protein mixture. More than 10 mg of protein could be used because the maximum amount of protein adsorbed on 1 g of glass is *ca.* 80 mg. If a larger column is used, more proteins should be separated. This method might not be applicable to the fine separation of proteins but rather to the large-scale preparation of proteins.

The surfaces of non-coated glass are labile and enzymes bound on non-coated glass for a long time are inactivated on storage[10]. Enzymes adsorbed on the surfaces of coated glass are stable and the glass is useful as a support for the immobilized enzymes. Hemoglobin adsorbed on coated glass is not eluted with 10% ethanol, 10% butanol, 10% acetone, water saturated with octyl alcohol, 0.1 *M* sodium thiocyanate, or 7 *M* urea. The conditions for elution of hemoglobin or other proteins strongly adsorbed on coated glass require further investigation.

## CONCLUSION

Proteins were separated by adsorption chromatography on siliconized porous glass in a water medium. A mixture of bovine serum albumin and hemoglobin in 5 m*M* Tris–HCl (pH 7.6) was applied on a coated glass column. Albumin passed through the column with a recovery of 32% and was separated from hemoglobin, which was adsorbed on the column. The results of loading of bovine globulin in

0.01 *M* phosphate (pH 7.4) on the column showed that $\alpha_2$-globulin was eluted and $\beta$- and $\gamma$-globulins were adsorbed by hydrophobic bonding. Silicone-coated glass should be useful for the large-scale preparation of proteins or other substances in aqueous media.

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## Note

# Simultaneous quantitation of thioureas in rat plasma by high-performance liquid chromatography

HIROKO KOBAYASHI*, OSAMI MATANO and SHINKO GOTO

*Chemistry Division, Institute of Environmental Toxicology, Suzuki-cho 2-772, Kodaira, Tokyo 187 (Japan)*

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Certain thioureas have been reported to be carcinogenic and teratogenic in mammals. Thiourea (TU) and tetramethylthiourea are carcinogens toward rats and mice[1–3]. Methylthiourea and ethylthiourea have been found to have teratogenic effects in rats[4]. 1,3-Dimethylthiourea induced conjunctivitis and dermatitis of the eyelids of textile workers[5]. Ethylenethiourea, which is a degradation product of fungicidal ethylenebisdithiocarbamates, has carcinogenic and teratogenic properties in rats or mice[6–9]. For monitoring these toxic thioureas and related compounds in animals, a selective and effective method is required.

Current methods for determining TU and ETU include thin-layer chromatography (TLC)[10,11] and gas–liquid chromatography (GLC)[11–14].

This paper describes a superior method for the isolation, identification and determination of thioureas in rat plasma by high-performance liquid chromatography (HPLC) without derivatization.

## EXPERIMENTAL

### *Materials and reagents*

Thiourea (TU) was obtained from Kanto Chemical (Tokyo, Japan), methylthiourea (MeTU), ethylenethiourea (ETU) and 1,3-diethylthiourea (1,3-DETU) from Tokyo Chemical (Tokyo, Japan), ethylthiourea (EtTU) from ICN Pharmaceuticals (Plainview, NY, U.S.A.) and 1,3-dimethylthiourea (1,3-DMTU) from Nakarai Chemicals (Kyoto, Japan). 1,1-Dimethylthiourea (1,1-DMTU) was synthesized from dimethylammonium chloride and ammonium thiocyanate by the method mentioned below. A standard solution of thioureas was prepared in distilled water. HPLC-grade methanol was obtained from Wako (Osaka, Japan). All other reagents were of analytical-reagent grade. The silica gel used for column chromatography was Kieselgel 60 (0.063–0.200 mm, 70–230 mesh) from E. Merck (Darmstadt, G.F.R.).

### *Synthesis of 1,1-dimethylthiourea*

1,1-Dimethylthiourea was synthesized by modifying the method of Gebhart[15], which was devised for preparing 1-methyl-1-phenylthiourea.

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Ammonium thiocyanate (28.5 g, 0.375 mol) in distilled water (20 ml) was added to dimethylammonium chloride (20.25 g, 0.248 mol). The mixture was stirred for 50 h at 100°C, then rendered alkaline with 10% sodium hydroxide solution. The solution was extracted three times with 300 ml of ethyl acetate and the extract was washed three times with 25 m of distilled water. The organic phase was dried over anhydrous sodium sulphate and evaporated *in vacuo*. The residue was extracted with 100 ml of hot chloroform and, after evaporation to dryness, the residue was purified by recrystallization from ethanol to give 1,1-dimethylthiourea as colourless needles, m.p. 161°C, with the following properties: infrared, $\nu_{max.}^{KBr}$ 3390, 3280, 3190, 1625, 1545, 1420, 1365 $cm^{-1}$; UV, $\lambda_{max.}^{CH_3OH}$ 243 nm; mass spectrometry, *m/z* 104 ($M^+$, 100%), 90(0.2%), 88(2.9%), 76(3.0%), 74(0.3%), 73(2.1%), 71(6.1%), 60(21.1%), 44(63.8%); $^1$H-nuclear magnetic resonance ($C^2HCl_3$), $\delta$ 3.3 (6H, s, 2 × $CH_3$), 5.77 (2H, broad s, $NH_2$). Elemental analysis: calculated for $C_3H_8N_2S$, C 34.62, H 7.69, N 26.92; found, C 34.48, H 7.69, N 26.81.

*Apparatus*

All analyses were carried out using a Jasco-Tri-Rotal high-performance liquid chromatograph (Japan Spectroscopic, Tokyo, Japan) equipped with a Uvidec-100 spectrophotometer monitoring the absorbance at 240 nm. The column (25 cm × 4.6 mm I.D.) was packed with ODS SC-02 (Japan Spectroscopic) and eluted with 5% methanol in water at a flow-rate of 0.8 ml/min (48 $kg/cm^2$) and ambient temperature. The detector sensitivity was set at 0.256 or 0.032 a.u.f.s.

*Plasma*

Adult male Wistar rats were anaesthetized with diethyl ether. The blood was collected with heparinated syringe and then centrifuged at 3000 rpm for 5 min. The supernatant was used for study.

*Plasma extraction procedure*

Plasma (1–2 ml) was shaken vigorously for 10 min with 5 ml of ethanol. The mixture was centrifuged at 3000 rpm at room temperature for 20 min, then the organic layer was carefully transferred to a flask and evaporated to dryness under a stream of nitrogen. The residue was dissolved in a small volume of chloroform and applied to a 125 × 15 mm I.D. silica gel column (10 g). After the column had been washed with 20 ml of chloroform and with 20 ml of 3% methanol in chloroform, 1,3-DETU, ETU, 1,3-DMTU and 1,1-DMTU were eluted with 60 ml of 3% methanol in chloroform, and TU, MeTU and EtTU with 100 ml of 10% methanol in chloroform.

These fractions were concentrated to about 1 ml *in vacuo* at 40°C, and then blown to dryness with a stream of nitrogen. The residue was dissolved in 2 ml of mobile phase and this solution was injected into the HPLC system.

## RESULTS AND DISCUSSION

The wavelengths of maximal absorption for TU, MeTU, EtTU, 1,1-DMTU, 1,3-DMTU, 1,3-DETU, TMTU and ETU were 242, 240.5, 242.5, 243, 239, 242, 255.5 and 239.5 nm, respectively. For the simultaneous determination of these compounds, 240 nm was selected as a reasonable wavelength to monitor the chromatograms.

Normal- and reversed-phase partition systems were compared and the reversed-phase system (SC-02) with 5% methanol in water as the mobile phase at ambient temperature was found to be best for separating most of the thioureas.

The only exception was for TMTU, which gave a long retention time (112.0 min) and broad peak on the reversed-phase system. In contrast, a normal-phase system (SS-05) with 3% methanol in chloroform–*n*-hexane (80:20) as the mobile phase gave a sharp peak at a retention time of 4.5 min and was therefore suitable for the determination of TMTU.

A typical chromatogram obtained from a rat plasma sample is shown in Fig. 1. The retention times for TU, MeTU, ETU, EtTU, 1,3-DMTU, 1,1-DMTU and 1,3-DETU were 4.0, 5.2, 6.0, 8.9, 9.0, 11.2 and 40.6 min, respectively. Five of the tested compounds were clearly separated. However, EtTU and 1,3-DMTU could not be separated under these or any other conditions. However, these two thioureas can easily be separated by column chromatography on silica gel. When 3% methanol in chloroform was used as eluent in the silica gel column, ETU, 1,3-DMTU, 1,1-DMTU and 1,3-DETU could be eluted and separated from the other thioureas, which were eluted by 10% methanol in chloroform (Fig. 2).

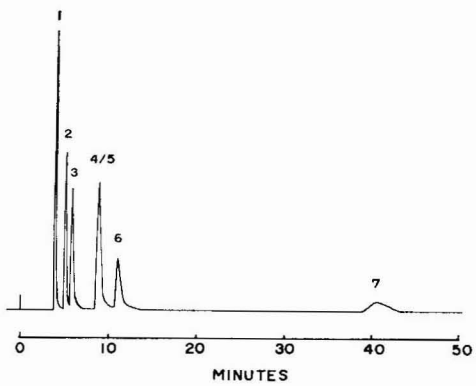


Fig. 1. Chromatogram of a standard mixture of thioureas. Column, SC-02 (25 cm × 4.6 mm I.D.); mobile phase, water–methanol (95:5); flow-rate, 0.8 ml/min; temperature, ambient; wavelength of detection, 240 nm; sensitivity, 0.256 a.u.f.s.; amounts injected, 200 ng of each compound. Peaks: 1 = TU; 2 = MeTU; 3 = ETU; 4 = EtTU; 5 = 1,3,-DMTU; 6 = 1,1-DMTU; 7 = 1,3-DETU.

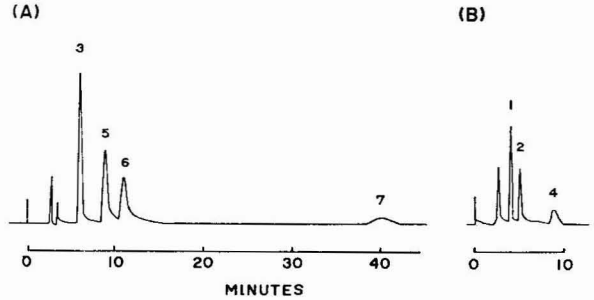


Fig. 2. Chromatogram of plasma spiked at 1 ppm. Operating conditions and peaks as in Fig. 1, except 0.032 a.u.f.s.; (A) 3% methanol in chloroform fraction, 50 ng of each compound injected; (B) 10% methanol in chloroform, 25 ng of each compound injected.

Calibration graphs were prepared for TU, MeTU, EtTU, ETU, 1,1-DMTU, 1,3-DMTU and 1,3-DETU by plotting the peak height or peak area against amount, as illustrated in Fig. 3. Linear relationships were obtained in the range 15–200 or 100–400 ng for each compound.

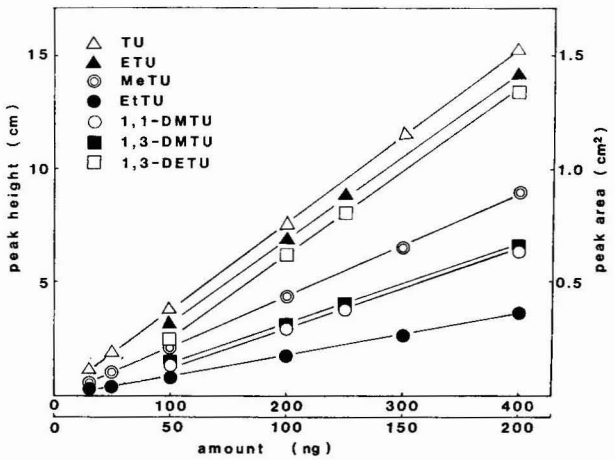


Fig. 3. Calibration graphs for determination of thioureas. Range of amounts: 15–200 ng for TU, MeTU and EtTU; 100–400 ng for ETU, 1,3-DMTU, 1,1-DMTU and 1,3-DETU. Peak area was used only for 1,3-DETU.

Table I gives the recovery data obtained for the plasma. The thioureas were recovered in high yield at concentrations from 1 to 100 ppm.

The detection limits were 0.05 ppm for 1,1-DETU and 0.02 ppm for the others (sample, 2 ml; injection volume, 100 $\mu$l).

TABLE I

RECOVERY OF THIOUREAS

| *Amount added (ppm)* | *Recovery (%)** | | | | | | |
|---|---|---|---|---|---|---|---|
| | *TU* | *MeTU* | *ETU* | *EtTU* | *1,3-DMTU* | *1,1-DMTU* | *1,3-DETU* |
| 100 | 84 | 92 | 81 | 74 | 100 | 84 | 82 |
| 25 | 88 | 89 | 91 | 88 | 90 | 85 | 96 |
| 10 | 75 | 70 | 79 | 84 | 90 | 71 | 78 |
| 5 | 93 | 94 | 78 | 89 | 99 | 80 | 86 |
| 2.5 | 92 | 93 | 79 | 86 | 97 | 74 | 92 |
| 1 | 91 | 76 | 84 | 73 | 89 | 77 | 78 |
| Mean | 87 | 86 | 82 | 82 | 94 | 79 | 85 |

* Results are the means of duplicate determinations.

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## Note

# Chromatographic separation of some biogenic amines on a weakly acidic ion-exchange resin

TOKUICHIRO SEKI
*College of Biomedical Technology, Osaka University, Toyonaka, Osaka 560 (Japan)*
(Received November 4th, 1980)

Chromatographic separation of biogenic amines has been performed by ion-exchange chromatography[1–7], reversed-phase partition chromatography and paired-ion chromatography[8–10]. These methods provide a good separation of catecholamines and their metabolites; however, a simultaneous separation of basic metabolites of catecholamines and serotonin by means of isocratic elution has not been reported.

We have found that catecholamines can be eluted isocratically from a column of Amberlite IRC-50 with a buffer of pH 4. Various buffers were tried as eluents, and separation of catecholamines, octopamine, 3-O-methylated catecholamines and serotonin was achieved with a buffer of pH 4.4 containing propionate (0.15 *M*), tartrate (0.10 *M*), EDTA (0.002 *M*) and boric acid (0.35 *M*) as the eluent.

## CHROMATOGRAPHIC SYSTEM

Amberlite IRC-50 ($Na^+$; particle size 50–60 $\mu$m) was buffered at pH 4.4 and washed with the eluent and packed into a chromatographic tube (0.8 cm I.D.) equipped with a column adjuster. The eluent was pumped into the column at a flow-rate of 1.0 ml/min (a constant delivery pump, Jasco Model LCP-150) at 50°C, and the final length of the column was 24 cm. The sample was dissolved in the eluent and 1.0 ml of the solution was added to the column using a loop injector (Kyowa Seimitsu, sampler, Model M2). Amines in the eluate were monitored fluorometrically (spectrofluorometer, Jasco Model FP-4) with excitation at 285 nm and emission at 325 nm.

## RESULTS AND DISCUSSION

The elution pattern is shown in Fig. 1. Amines were eluted in the order of decreasing polarity. Propionic acid was incorporated in the eluent in order to reduce non-ionic adsorption of amines on the resin. Separation of dopamine and octopamine was possible only when both tartrate and boric acid were present in the eluent. Reduction of retention time of catecholamines by the use of the eluent seemed to be due to the formation of a negatively charged catecholamine–borate–tartrate complex at pH 4.4. At higher pH, separation of dopamine from octapamine could be improved, but separation of norepinephrine from epinephrine became worse. The elution pattern was quite reproducible and the column could be used repeatedly.

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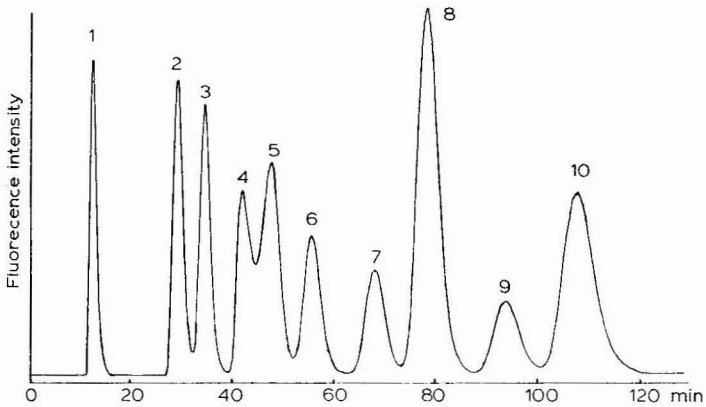


Fig. 1. Elution of standard samples of various amines. Peaks: 1 = dopa; 2 = norepinephrine; 3 = epinephrine; 4 = dopamine; 5 = octopamine; 6 = normetanephrine; 7 = metanephrine; 8 = tyramine; 9 = 3-methoxytryamine; 10 = serotonin. Column size, 24 × 0.8 cm I.D.; column temperature, 50°C; flow-rate of the mobile phase, 1.0 ml/min.

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## Book Review

*Recent developments in chromatography and electrophoresis, 10* (Proc. 10th Int. Symp. Chromatography and Electrophoresis, Venice, June 19–20, 1979; *Analytical Chemistry Symposia Series*, Vol. 3), edited by A. Frigerio and M. McCamish, Elsevier, Amsterdam, Oxford, New York, 1980, X + 342 pp., price Dfl. 140.00, US$ 68.25, ISBN 0-444-41871-7.

This is the third volume of a set that was commenced with the title *Chromatography Symposia Series*. The theme has presumably been widened in order to accommodate the electroanalytical title of Vol. 2.

The present volume is a collection of the 34 papers presented at the *10th International Symposium on Chromatography and Electrophoresis* held in June 1979 and may be related to a similar collection from the 9th Symposium and published in Vol. 1. As was the case on that occasion most forms of chromatography and electrophoresis are included. Thus, there are gas chromatography in its more ordinary and capillary forms, high-performance liquid chromatography, high-performance thin-layer chromatography and ordinary thin-layer chromatography, isoelectric focusing, polyacrylamide gel electrophoresis, amino acid analysers, etc.

As in the case of its earlier companion, there is a distinct pharmaceutical and biochemical flavour, and there are two main divisions, namely areas of use and technique. For the former there are sections on drug analysis (9 papers), analysis of endogenous compounds (7 papers) and environmental studies (3 papers), with electrophoresis (5 papers), fluorometry (5 papers), and instrumental developments (5 papers) being the sections devoted to technique. The papers generally deal with the observations and results of individual projects and are set out in conventional scientific paper style rather than of a review type. However, the paper on recent trends in isoelectric focusing deviates from this pattern, and although the same authors (Righetti, Gianazza and Bosisio) reviewed the biochemical and clinical applications of isoelectric focusing in Vol. 1, they give adequate reasons for this second review.

For advances in isoelectric focusing (IEF), Righetti *et al.* give significance to the possibilities of agarose IEF, focusing of peptides, two-dimensional IEF–electrophoresis for ligand–protein and protein–protein interactions, etc. as well as describing advances in set areas, such as carrier ampholytes, analytical chambers, preparative approaches and methodology.

By being essentially a collection of individual papers on projects, this book will be less interesting than Vol. 1 to the reader wishing to have a general overview of the state of development of the various subject areas. However, other readers will be interested in the specific areas covered and in this respect it is appropriate to note that phenolic compounds, food dyes, cefuroxime in biological samples, ergot alkaloids, the presence in plasma of isosorbide nitrates, acetylsalicylic acid and mefloquine, peptides, proteins, steroids, bile acids, lipids, aromatic hydrocarbons, etc. are discussed in terms of separation and, frequently, of quantitation. Some attention is given

to preliminary clean-up procedures where this may be necessary. In many cases there is consideration of variations in technique, such as that of the comparison of polar and non-polar silicone stationary phases in the gas–liquid chromatographic separation of drugs.

Separation is, of course, the purpose of chromatographic and electrophoretic methods, but it is frequently necessary to use complementary methods when components are not readily resolved. This may be by derivatisation, the use of specific visualising agents or other methods of specific detection. The nitrogen–phosphorus selective detector (NPD) is a recent development. The paper on the gas chromatographic analysis of hydroxy steroids and fatty acids gives an interesting account of the role of bis(N,N-dialkylamino)dimethylsilane for derivatising in order to permit analysis with the NPD. In another contribution, bile acids in serum are separated by capillary gas chromatography following a preliminary extraction and clean-up procedure and conversion to their methyl ester trimethylsilyl ether derivatives.

The papers on post-column derivatisation in order to exploit fluorescence detection are worthwhile and critical in their approach. They set the tone of the section devoted to fluorescence by being the main justification for a separate section devoted to this theme. Preliminary treatment and sample collection are necessary adjuncts to chromatographic procedures and to any analytical method, but it is doubtful whether papers devoted solely to such aspects have their real place in a Symposium devoted to another stage of the analytical process. There are a few here and the paper on the determination of methanol, ethanol, benzene and cyclohexane in air using charcoal as solid sorbent falls clearly into this category.

The instrumental section is rather scrappy, being made up of papers on pressurised thin-layer chromatography, a low-pressure liquid chromatography pump, photometric densitometric evaluation of thin-layer chromatograms, chemical modification of silica gels and aspects of the application of capillary gas chromatography. Such a criticism is often inevitable in compilations of papers presented at conferences and symposia and cannot be avoided unless the presentations are systematically selected beforehand.

The camera-ready typing is evenly produced to give the book a pleasing appearance and the diagrams are clear and easy to follow. The book will doubtless find its way to library shelves but the price compared with that of Vol. 1 published a year earlier is 16.6% greater in Dutch currency and 28.2% greater in US$ for 15 fewer pages. Inflation rates of the British economy are not so outrageous after all!

*Cardiff (Great Britain)* J. D. R. THOMAS

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## Book Review

Two short introductions to thin-layer chromatography: *Thin layer chromatography, a laboratory introduction*, by P. Jenks and P. Wall, BDH, Poole, 1980, 46 pp., price £1.95 (Great Britain), £3.00 (export); and *Thin layer chromatography*, by W. Götz, A. Sachs and H. Wimmer, BDH, Poole, 1980, 114 pp., price £4.00 (Great Britain), £6.00 (export).

The first of these two booklets tries to answer technical queries posed by the many customers of BDH Chemicals Ltd., by "*ab initio*" chromatographers. As such it is a let-down. It has a 25-line introduction, leaving the reader with the impression that first there was Tswett and Day and in the later 1930s thin-layer chromatography (TLC) was started and became serious in 1958. Two lines would have sufficed to mention that TLC was a logical outcome of paper chromatography (PC) and that a wealth of data suitable for TLC can be found in the PC literature. But these lines were not written...

The authors use a confusing terminology, *e.g.*, referring to "TLC sorbents" for all forms of TLC. They give a number of experiments to be carried out. Some seem pointless, such as a two-dimensional TLC of dyes, separating only four substances. Surely there are better examples! The reference list mentions two books by "Fiegl" (is this a mixture of the Austrian Prime Minister Figl and the chemist Feigl?), and it cites Randerath's book twice (once the first and once the second edition). To sum up: I would not recommend this text to "*ab initio*" chromatographers.

The second book is an excellent introduction to the use of TLC in clinical chemistry. However, it would have profited by suitable reference to the pioneering work of C. E. Dent, Ivor Smith, Ian Bush and many others who laid the basis of clinical applications. But they used paper chromatography... Both volumes are illustrated with excellent colour plates.

*Lausanne (Switzerland)* MICHAEL LEDERER

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## 8. SUBSTANCES CONTAINING HETEROCYCLIC OXYGEN

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## 19. PROTEINS

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See also 237.

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## 20. ENZYMES

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*20h. Isomerases*

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See 1989.

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Vol. 207, No. 2

journal of chromatography news section

## NEW PRODUCTS

N-1549

REVERSED-PHASE HPLC COLUMN

Hamilton has designed a reversed-phase HPLC column for application where pH and salt concentrations are important. The new Hamilton PRP-1 column is said to be ideal for the separation of polar compounds. The hydrophobic qualities are said to be equal to or better than any $C_{18}$ silica.

Hamilton's PRP-1 is filled with rigid 10-$\mu$m macroporous resin spheres which can selectively adsorb both ionic and neutral solute species. These non-swelling adsorbents operate over a relatively broad pH range. The physical stability precludes the need for a guard column or concern for damage to the packing.

N-1550

IEF CALIBRATION KITS

The three Pharmacia isoelectric focusing p*I* calibration kits contain ten vials of pre-weighed lyophilized mixtures of 8–11 purified proteins. The proteins focus as distinct bands of known p*I*'s enabling the user to measure pH gradients and determine isoelectric points. The first kit covers a broad pH range from 3 to 10. The second one offers a low range, from 2.5 to 6.5. The third calibration kit covers a higher pH range, from 5 to 10.5.

N-1546

PURE WATER

Organic-free water is now available from J.T. Baker Chemicals BV. This type of pure water, called "Baker Instra-Analyzed" water, is suitable for various trace organic analyses, including: total organic carbon (TOC), for use as a standard, and trihalomethane (THM) analysis, for the preparation/dilution of standards. The TOC content in "Baker Instra-Analyzed" water is less than 100 ppb and has been reported by water authorities to be as low as 40 ppb. The total THM levels in "Baker Instra-Analyzed" water are less than 0.1 ppb. The water is available in 3.75-l pre-cleaned bottles.

N-1547

TLC MEDIUM

Chromatronix has introduced a thin-layer chromatography medium that separates by ion exchange. This product, Fixion™, is said to have a high sample throughput and many samples can be run at one time. The Fixion medium can handle samples with high salt content. Fixion has a thin, densely packed bed, made up of spherical particles of polystyrene–divinylbenzene copolymer. The mean particle diameter is 8 $\mu$m.

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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.

NEW ANALYTICAL PRODUCT GROUP

Erba Instruments, Inc., a newly formed marketing and service organization representing Carlo Erba, internationally known manufacturer of chromatographic and analytical instruments as well as accessories, displayed the following Carlo Erba products at this year's Pittsburgh Conference: high resolution gas chromatograph Model FV 4160, high resolution gas chromatograph Model FV 2900, elemental analyzer Model CHN+OS 1106, automatic nitrogen analyzer Model ANA 1400 and mercury pressure porosimeter Model PO 200.

Major offices of the new firm are headquartered on the East and West Coasts with technically staffed service offices located throughout the United States. The West Coast headquarters will be directed by Mr. Ben Apon, 12015 Slauson Avenue, Suite F, Santa Fe Springs, CA 90670, U.S.A. The East Coast headquarters will be directed by Mr. Tom Jackson, Northway Office Park, 3 Dearborn Rd., Peabody, MA 01930, U.S.A.

NEW SALES AND SERVICE CENTRE

On January 20, 1981, Spectra-Physics opened its new European Sales and Service Centre, located at Siemensstrasse 20, D-6100 Darmstadt-Kranichstein, G.F.R., Tel. (06151) 708-0.

## NEW BOOKS

**Halogenated biphenyls, terphenyls, naphthalenes, dibenzodioxins and related products**, edited by R.D. Kimbrough, Elsevier/North-Holland Biomedical Press, Amsterdam, New York, 1980, XX + 506 pp., price Dfl. 195.00, US$ 95.00, ISBN 0-444-80253-3.

**Dictionary of chemical terminology**, edited by D. Kryt, Elsevier, Amsterdam, Oxford, New York, 1980, XII + 600 pp., price Dfl. 195.00, US$ 95.00, ISBN 0-444-99788-1.

**Introduction in colloid and surface chemistry**, by D.J. Shaw, Butterworths, London, 3rd ed:, 1980, 272 pp., price £ 5.95, US$ 15.00, ISBN 0-408-71049-07.

**Analysis of nucleic acid constituents**, by C.T. Wehr, Varian, Palo Alto, CA, 1980, price US$ 10.00.

**Chemische Reaktionsdetektoren für die schnelle Flüssigkeits-Chromatographie**, by G. Schwedt, Hüthig, Heidelberg, 1981, 213 pp., price DM 38.00, ISBN 3-7785-0687-0.

**Pesticide analytical methodology**, edited by J. Harvey, Jr. and G. Zweig, American Chemical Society, Washington, DC, 1980, X + 406 pp., US$ 38.00, ISBN 0-8412-0581-7.

**Size exclusion chromatography (GPC)**, edited by T. Provder, American Chemical Society, Washington, DC, 1980, VIII + 312 pp., US$ 30.75, ISBN 0-8412-0586-8.

**Tumours that secrete catecholamines: their detection and clinical chemistry**, by R. Robinson, Wiley, Chichester, New York, 1980, ca. 148 pp., price US$ 40.00, £ 14.00, ISBN 0-471-27748-7.

**Polyamines in biomedical research**, by J.M. Gaugas, Wiley, Chichester, New York, 1980, ca. 512 pp., price US$ 85.00, £ 30.00, ISBN 0-471-27629-4.

**Qualitative analysis of flavor and fragrance volatiles by glass capillary gas chromatography**, by W. Jennings and T. Shibamoto, Academic Press, New York, 1980, VIII + 472 pp., price US$ 39.00, ISBN 0-12-384250-6.

**Analytiker Taschenbuch, Band II**, edited by R. Bock, W. Fresenius, H. Günzler, W. Huber, and G. Tölg, Springer, Berlin, Heidelberg, New York, 1981, ca. 360 pp., price DM 78.00, US$ ca. 45.90, ISBN 3-540-10338-4.

**Fats and oils: chemistry and technology**, edited by R.J. Hamilton and A. Bhati, Applied Science, Barking, 1981, XII + 263 pp., price £ 24.00, ISBN 0-85334-915-0.

**Electron transfer reactions**, by R.D. Cannon, Butterworths, London, 1980, 368 pp., price £ 32.00, US$ 80.00, ISBN 0-408-10646-8.

**Ullmanns Encyklopädie der technischen Chemie, Band 5, Analysen- und Messverfahren**, edited by H. Kelker, Verlag Chemie, Weinheim, Deerfield Beach, Basel, 4th ed., 1980, XVI + 1010 pp., price SFr 670.00, ISBN 3-527-20005-3.

**Nondestructive activation analysis – with nuclear reactors and radioactive neutron sources**, edited by S. Amiel, Elsevier, Amsterdam, Oxford, New York, 1981, XVI + 364 pp., price Dfl. 170.00, US$ 83.00, ISBN 0-444-41942-X.

J.F.K. HUBER RECEIVES THE DAL NOGARE AWARD

Professor Josef Franz Karl Huber has received the Dal Nogare award from the Chromatography Forum of the Delaware Valley on Tuesday, March 10, 1981, at the Pittsburgh Conference in Atlantic City, NJ, U.S.A. This was the tenth anniversary of the award which is given annually by the Chromatography Forum for significant contributions to chromatographic theory, instrumentation and applications. J.F.K. Huber received the award at a special tenth anniversary symposium which honored the previous awardees, many of whom presented papers at that time.

## COURSES

**Finnigan Institute** has scheduled the following courses for April–June 1981.
April 6–10: Using GC–MS in the Compliance and Enforcement Context: A Legal/Technical Assessment, US$ 295.00; April 6–10: Liquid Chromatography: Basic Concepts and Techniques, US$ 750.00; April 13–17: Basic GC–MS–DS, US$ 750.00; April 21–24: Chemical Derivatization, US$ 600.00; May 4–8: Metabolism and Pharmacokinetics: Quantitative and Qualitative Analysis, US$ 750.00; May 11–15: Atomic Absorption: Basic Concepts and Techniques, US$ 750.00; May 11–15: Analysis of Priority Pollutants by GC–MS, US$ 750.00; May 18–22: Gas Chromatography: Basic Concepts and Techniques, US$ 750.00; June 1–3: Analytical Pyrolysis by Eugene Levy, US$ 450.00; June 8–12: Introduction to GC–MS; Basic Mass Spectral Interpretation, US$ 575.00; June 8–12: Liquid Chromatography: Basic Concepts and Techniques, US$ 750.00; June 15–16: Mass Spectral Interpretation: Applications, US$ 300.00; June 22–26: Analysis of Priority Pollutants by Chromatographic Techniques, US$ 750.00; June 29–July 3: Advanced Atomic Absorption, US$ 750.00. For more information, contact Nancy Kranpitz, Finnigan Institute, 11 Triangle Park Drive, Cincinnati, OH 45246, U.S.A. Tel. (513) 772-5500.

The Department of Chemistry of **Loughborough University of Technology**, will hold the following courses during 1981. March 23–27: Gel Filtration and Electrophoresis; April 6–10: Gas Chromatography; July 6–10: High Performance Liquid Chromatography. For each course the fee is £ 170, including residence and all meals. Further details are available from:
Miss J.M. Brown, Department of Chemistry, Loughborough University of Technology, Loughborough, Leicestershire LE11 3TU, Great Britain.

**Checking Foodstuffs for Trace-Organics** (workshop course), Guildford, July 13–17, 1981. Strategies for a range of analytes including mycotoxins, residues, nitrosamines, additive and packaging contaminants will be considered, e.g. in connection with processed foods and market produce. Information from Dr. E. Reid, Wolfson Bioanalytical Unit, Robens Institute, University of Surrey, Guildford GU2 5XH, Great Britain.

## MEETING

SYMPOSIUM ON PRACTICAL ASPECTS OF HPLC

A symposium on practical aspects of modern HPLC will be organised by the H. Knauer GmbH in Berlin in the first half of November 1981.

Papers are invited dealing with the practical aspects of HPLC in the following fields: pharmaceutical, organic, inorganic and physical chemistry.

It is envisaged that the symposium will last for two days, the papers being presented in prallel lecture and poster sessions.

Fur further information, registration and submission of papers, please contact: Dr. I. Molnár, Wissenschaftliche Gerätebau Dr. Ing. H. Knauer GmbH, Hegauer Weg 38, D-1000 Berlin 37, G.F.R.

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## PUBLICATION SCHEDULE FOR 1981

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

| MONTH | N 1980 | D 1980 | J | F | M | A | M | J | J | A | S | O | N | D |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Journal of Chromatography | | | 203<br>204<br>205/1<br>205/2 | 206/1<br>206/2<br>206/3 | 207/1<br>207/2<br>207/3 | 208/1<br>208/2<br>209/1 | 209/2 | The publication schedule for further issues will be published later. | | | | | | |
| Chromatographic Reviews | | | | | | | 220/1 | | | | | | | |
| Biomedical Applications | 221/1 | 221/2 | 222/1 | 222/2 | 222/3 | 223/1 | 223/2 | | | | | | | |

## INFORMATION FOR AUTHORS

(Detailed *Instructions to Authors* were published in Vol. 193, No. 3, pp. 529–532. A free reprint can be obtained by application to the publisher)

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

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

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

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

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

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# Preparation of Catalysts II

## Scientific Bases for the Preparation of Heterogeneous Catalysts

Proceedings of the Second International Symposium, Louvain-la-Neuve, Belgiu
4-7 September, 1978

**B. DELMON, P. GRANGE, P. JACOBS** *and* **G. PONCELET** *(Editors)*

***Studies in Surface Science and Catalysis, 3***

This conference was organized around two specific unit processes: impregnation a
activation, and was particularly concerned with the chromatographic effect, tra
port in pores, calcination, activation by reduction and sulfidation, carrier effects a
compound transformation. New aspects in the preparation of real i.e. industrial ca
lysts were also discussed.

Centred around four plenary lectures and three extended communications, the bo
discusses: preparation and pretreatment of mixed metal oxides and of metal or me
oxide supported catalysts, by (co)-impregnation, (co)-precipitation, ion exchange,
sorption, and other methods with particular reference to the preparation conditio
These include drying, crystallite size distribution, chromatographic processes, in
action with and influence of the support, dispersion and distribution of the act
components, stabilization, transport reactions, etc. In addition to the plenary lectu
44 communications are included. At a half-day 'mini-symposium,' the normalizat
of methods for catalyst characterization was discussed.

**PLENARY LECTURES:**

- **The design of catalysts *(D. L. Trimm).***
- **Separation of catalysts by adsorption of metal complexes on mineral oxides *(J. P. Brunn***
- **Catalyst activation by reduction *(N. Pernicone and F. Traina).***
- **Preparation and properties of monodispersed colloidal metal hydrous oxides *(E. Matije***

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