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Separation of polycyclic aromatic hydrocarbons on a wide-pore polymeric C₁₈ bonded phase

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Abstract

This paper presents a novel wide-pore C₁₈ bonded phase. This bonded phase has polymeric properties and can separate a broad range of polycyclic aromatic hydrocarbons (PAHs) in different matrices. A column selectivity test mixture containing three well known chosen PAHs was used to evaluate the selectivity ($\alpha_{\text{TBN/BaP}}$ value) of the bonded phase under sub-ambient, ambient and elevated temperatures. The variation of the column selectivity with temperature was studied. The results showed that the separation of critical PAH isomers increased with decreasing temperatures. The separation of the 16 US Environmental Protection Agency PAH priority pollutants was found to vary continuously with temperature and $\alpha_{\text{TBN/BaP}}$ value. The effect of the slope of the gradient was also investigated and it was proved that a steeper gradient increased the separation of dibenz[*a,h*]anthracene and benz[*ghi*]perylene. Due to the fact that the column has a relative small internal diameter (3.0 mm), reduction of the mobile phase consumption was found to be almost 50% compared to the standard-size (4.6 mm) HPLC columns.

1. Introduction

Polycyclic aromatic hydrocarbons (PAHs) are environmental pollutants formed at high temperatures and under pyrolytic conditions during the incomplete combustion of organic matter. They are found in air, water and soil. Sources of PAHs are widespread including coal/oil-burning power plants, coke oven plants and exhaust gases from traffic. The World Health Organization has issued a guide value maximum of 10 mg/ml for the single PAH benzo[*a*]pyrene [1].

Because certain PAH isomers exist and because many of these PAHs are toxic or even

carcinogenic/mutagenic [2], the development of an accurate and sensitive separation method is needed. Recommended analytical procedures are documented or proposed in several USA and European guidelines including the US Environmental Protection Agency (EPA) methods [3-5], International Standard Organization (ISO) method [6], German Standard (DIN) method [7] and Dutch Standard (NEN) method [8].

The EPA methods comprises a list of 16 PAH priority pollutants; the German and Dutch standard methods only specify 6 and 10 PAHs, respectively.

Because more and more PAHs are identified and investigated on their toxicity it can be expected that the list will be extended in the near

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future. All of these methods specify reversed-phase chromatography using octadecyl (C_{18}) bonded phases in combination with either fixed or wavelength-programmed ultraviolet (UV) and fluorescence (FL) detection techniques.

Several extraction techniques including liquid-liquid extraction (LLE) and solid-phase extraction (SPE) procedures [9], are used to clean-up or/and pre concentrate low levels of PAHs in various

matrices. SPE is recommended in EPA method 550.1 using either C_{18} cartridges or extraction disks [3]. It is well known that differences in retention and selectivity for the separation of this important group of compound exist among commercial available C_{18} columns [10]. These differences result from variation in a number of parameters in the manufacturing of the bonded phase, including alkyl chain length [11], phase coverage [12,13], pore size [14] and the type of surface modification chemistry employed [15]. Selectivity differences among "PAH columns" have been reported by a number of workers [16–18]. These differences have been pronominal related to the nature of the bonded phase. Bonded phases that are prepared using silane modification can be produced in two different ways depending on the reagents and reaction conditions used in the bonded phase synthesis resulting in either monomeric or polymeric stationary phases [19]. Column selectivity is found to vary continuously with temperature, regardless of the type of bonded phase used [20]. A simple test with three carefully chosen PAH solutes {phenanthro[3,4-*c*]phenanthrene (PhPh), 1,2:3,4:5,6:7,8-tetrabenzonaphthalene (TBN) and benzo[*a*]pyrene (BaP) } can be used to discriminate between both phase types [21]. This test also provides useful information about the selectivity towards critical PAH pairs. This paper presents a novel wide-pore polymeric C_{18} bonded phase which can be used for the separation of a broad range of PAHs. The separation of both the six Borneff PAHs and the already stated 16 priority pollutants will be demonstrated using either isocratic or gradient elution. Also the effect of the column temperature and the slope of the gradient will be discussed. The

differences in selectivity will be shown on the basis of chromatograms obtained from wide-pore polymeric C_{18} prepared with different $\alpha_{TBN/BaP}$ values.

2. Experimental

2.1. Chemicals

HPLC-gradient grade acetonitrile and water were from J.T. Baker (Deventer, Netherlands). Standard reference material (SRM) 1647c consists of an acetonitrile solution of 16 PAHs identified by the EPA as priority pollutants. SRM 869 was used to perform a selectivity test and contains BaP, TBN and PhPh. SRM 1597, a neutral, combustion-related mixture of PAHs isolated from coal tar is used to evaluate the performance of the chromatographic column. All SRMs were obtained from the National Institute of Standards and Technology (NIST, Gaithersburg, MD, USA) and were diluted to appropriate concentrations using HPLC-grade solvents. The six Borneff PAHs including perylene were used from a premixed (7 PAH mixture) standard (J.T. Baker).

2.2. Instrumentation

The HPLC gradient system (Perkin-Elmer, Norwalk, CT, USA) consisted of a Model LC-250 binary pump, a Model LC-290 UV-Vis variable-wavelength spectrophotometer and a Model LC 240 fluorescence detector. Data from this system were collected and evaluated using a Model 1020 LC integrator or a Model BD 111 flatbed recorder (Kipp and Zonen, Delft, Netherlands). Injections were done using a Model 7125 injector (Rheodyne, Cotati, USA) with a 20- μ l sample loop. The HPLC column (Bakerbond PAH-16 Plus, 250 \times 3.0 mm I.D. + integrated guard column, 20 \times 3.0 mm I.D.) was filled with wide-pore (300 Å) polymeric C_{18} with a particle size of 5 μ m (J.T. Baker).

A Mistral column thermostat (Spark Holland, Emmen, Netherlands) was used to obtain reproducible data between 10 and 45°C.

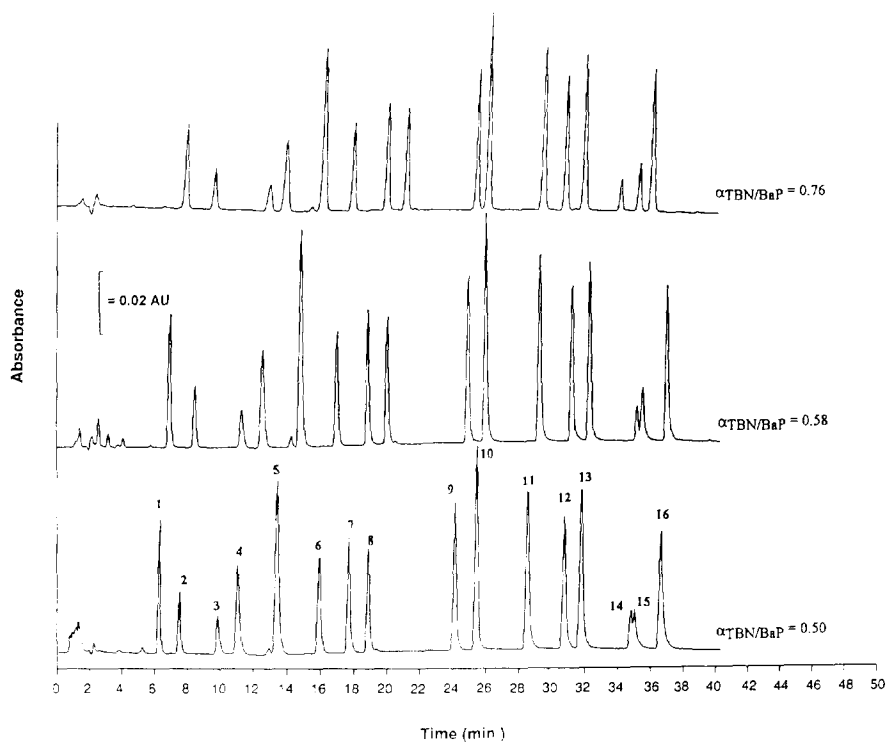


Fig. 3. Separation of SRM 1647c on three different batches Bakerbond wide-pore (300 Å) C_{18} bonded phase with different $\alpha_{TBN/BaP}$ values. Conditions: column: 250 mm \times 3.0 mm I.D., 5- μ m particles; mobile phase: acetonitrile–water (50:50) during 5 min, then linear gradient to 100% acetonitrile in 25 min at a flow-rate of 0.5 ml/min; temperature: $25 \pm 1^\circ\text{C}$; UV detection at 254 nm; injection of 10 μ l SRM 1647c [two-fold dilution in acetonitrile–water (50:50, v/v)]. Peaks: 1 = naphthalene (9.98 $\mu\text{g/ml}$); 2 = acenaphthalene (7.71 $\mu\text{g/ml}$); 3 = acenaphthene (10.28 $\mu\text{g/ml}$); 4 = fluorene (2.38 $\mu\text{g/ml}$); 5 = phenanthrene (1.74 $\mu\text{g/ml}$); 6 = anthracene (0.40 $\mu\text{g/ml}$); 7 = fluoranthene (3.83 $\mu\text{g/ml}$); 8 = pyrene (4.26 $\mu\text{g/ml}$); 9 = benz[*a*]anthracene (2.04 $\mu\text{g/ml}$); 10 = chrysene (1.85 $\mu\text{g/ml}$); 11 = benzo[*b*]fluoranthene (2.10 $\mu\text{g/ml}$); 12 = benzo[*k*]fluoranthene (2.34 $\mu\text{g/ml}$); 13 = benzo[*a*]pyrene (2.46 $\mu\text{g/ml}$); 14 = dibenz[*a,h*]anthracene (1.80 $\mu\text{g/ml}$); 15 = benzo[*ghi*]perylene (1.84 $\mu\text{g/ml}$); 16 = indeno[1.2.3-*cd*]pyrene (2.15 $\mu\text{g/ml}$).

phases can be prepared in two distinct ways, either by adsorbing a known quantity of water onto dry silica before reacting with the polyfunctional silane or in which the water is added directly into the reaction slurry to initiate polymerisation reaction. In the first method polymerisation of the silane is initiated by the adsorbed water at the silica surface. Bonded phases that are prepared using trifunctional silanes in the presence of water results in the formation of silane silanols, which can subsequently react with other silane molecules (cross-linking reactions) to form a silane polymer structure as illustrated in Fig. 1. The resulting hydrophobic surfaces are known to be hydrolytically stable [23]. For every

silanol group that disappears during the reaction, two new ones are potentially formed once the product is brought in contact with water. These silanol groups can subsequently be removed (end-capped) by reaction with a small monofunctional silane, e.g. trimethylchlorosilane (TMCS). Higher retention and slightly better resolution of compounds were obtained with end-capped polymeric C_{18} bonded phases. The access of small polar molecules to any “free” silanols within or underlying such polymer bonded phase has been shown to be lower than for bonded phase prepared from monofunctional silanes [24]. Consequently, the rate of equilibration of a polymer phase with mobile phase is more rapid than with

a monomeric phase [25]. Some important physical properties of the wide-pore polymeric C_{18} bonded phase, used in this study, are illustrated in Table 1. It is important to control the ligand density, because this property may influence the chromatographic selectivity and binding capacity of the bonded phase. Shape selectivity for PAH isomers increases with increasing phase density [26]. The pore size of the bonded phase also plays an important role in the phase selectivity. On narrow-pore (<100 Å) polymeric bonded phases isomers may coelute, whereas on wide-pore (e.g. 300 Å) material complete resolution can be achieved [19].

The flow-rate of the mobile phase is proportional to the square of the column diameter [27]. This means that the mobile phase consumption can be reduced by half when using a medium-bore (3.0 mm I.D.) column instead of one of 4.6 mm I.D. (standard-size column).

When the sample size is limited; this is sometimes the case when small elution volumes are used to elute the PAHs during the sample preparation step; the use of narrower columns result in better sensitivity due to the fact that the minimum detectable mass (not concentration!) is lower.

3.2. Bonded phase PAH selectivity test

All wide-pore C_{18} batches are subjected (internal quality control) to a characterization method developed by Wise and May [12,28]. This test is based on the retention of three carefully chosen PAH solutes as shown in Fig. 2. The test is intended primarily for predicting the potential selectivity for complex PAH mixtures and to discriminate between monomeric and polymeric type of C_{18} stationary phases. The selectivity factor $\alpha_{\text{TBN/BaP}}$ ($k'_{\text{TBN}}/k'_{\text{BaP}}$) correlates with the retention characteristic of PAHs and the bonded phase chemistry.

The elution order for polymeric type C_{18} should be PhPh, TBN and then BaP, as shown in Fig. 2. A classification scheme has been proposed, based on measurement of $\alpha_{\text{TBN/BaP}}$ values for experimental and commercial C_{18} columns [30]. Generally, column selectivity is enhanced

with lower $\alpha_{\text{TBN/BaP}}$ values. It is of extremely important to control the $\alpha_{\text{TBN/BaP}}$ during the C_{18} production. It should be noted that a small variation in this value will improve the precision of the PAH analysis. The $\alpha_{\text{TBN/BaP}}$ test is used by J.T. Baker to verify column reproducibility and assist in the adjustment of the bonding process. The influence of the $\alpha_{\text{TBN/BaP}}$ value is demonstrated in Fig. 3 in which the separation of SRM 1647c on three different batches (with variable $\alpha_{\text{TBN/BaP}}$ values) of wide-pore polymeric C_{18} is illustrated. Separation of all 16 PAHs can be achieved on bonded phases with $\alpha_{\text{TBN/BaP}}$ values between 0.6 and 0.8. At lower $\alpha_{\text{TBN/BaP}}$ values retention times of peaks 1–10 are decreased significantly. The resolution between the four-ring isomers benz[*a*]anthracene and chrysene is increased when the $\alpha_{\text{TBN/BaP}}$ value is decreased. The six-ring isomers benzo[*ghi*]perylene and indeno[1,2,3-*cd*]pyrene are better resolved at lower $\alpha_{\text{TBN/BaP}}$ values, but it should be noted that dibenz[*a,h*]anthracene and benzo[*ghi*]perylene will coelute or even reversed when bonded phases with too low $\alpha_{\text{TBN/BaP}}$ values are used.

3.3. Fluorescence programmed-wavelength detection of PAHs

Two of the difficulties in the analysis of PAHs are the low levels in which they have to be determined (low- to sub- $\mu\text{g/l}$ levels) and the relative complexity of the matrices of natural mixtures of PAHs from environmental extracts. Interfering compounds may coelute with the PAHs to be determined due to similar physical and chemical properties. Therefore a selective and sensitive detection technique is required. Fluorescence detection is the most sensitive and selective technique used for the measurement of PAHs in the picogram range. The EPA prescribes the use of both UV detection at 255 nm (for the first four PAHs) and fluorescence detection [excitation wavelength (λ_{ex}) 280 nm; emission wavelength (λ_{em}) > 390 nm with cut-off filter].

To apply maximum selectivity and sensitivity in quantification, the proper fluorescence excita-

tion and emission wavelengths must be chosen. Retention times must be perfectly reproducible and the baseline segment between two peaks must be long enough in order to change the $\lambda_{ex}/\lambda_{em}$ wavelength accurately.

It is well known that due to the lack of native fluorescence acenaphthylene must be detected with UV detection techniques (e.g. diode array detector).

The analysis of SRM 1647c and SRM 1597, which is a complex natural pyrolytic mixture of PAHs from coke oven tar, is depicted in Fig. 4 and demonstrates the use of wavelength-programmed fluorescence detection. The tar standard contains 12 certified PAHs and 18 non-certified PAHs/polycyclic aromatic compounds and was injected without any sample preparation procedure (only diluted a factor 200 with mobile

phase). Due to the high selectivity of both the stationary phase and the fluorescence detector 13 peaks were identified by comparison of the retention times. The first peak in the chromatogram comes from toluene that was used as dissolution solvent for the standard material. The excellent resolution of the stationary phase permits switching the $\lambda_{ex}/\lambda_{em}$ wavelengths 9 times which results in a very clean baseline.

3.4. Effect of the temperature

Reversed-phase separations are commonly carried out under ambient conditions, although temperature can have significant effect on the resolution of compounds in particular for PAHs [20].

The separation of six methylchrysene isomers

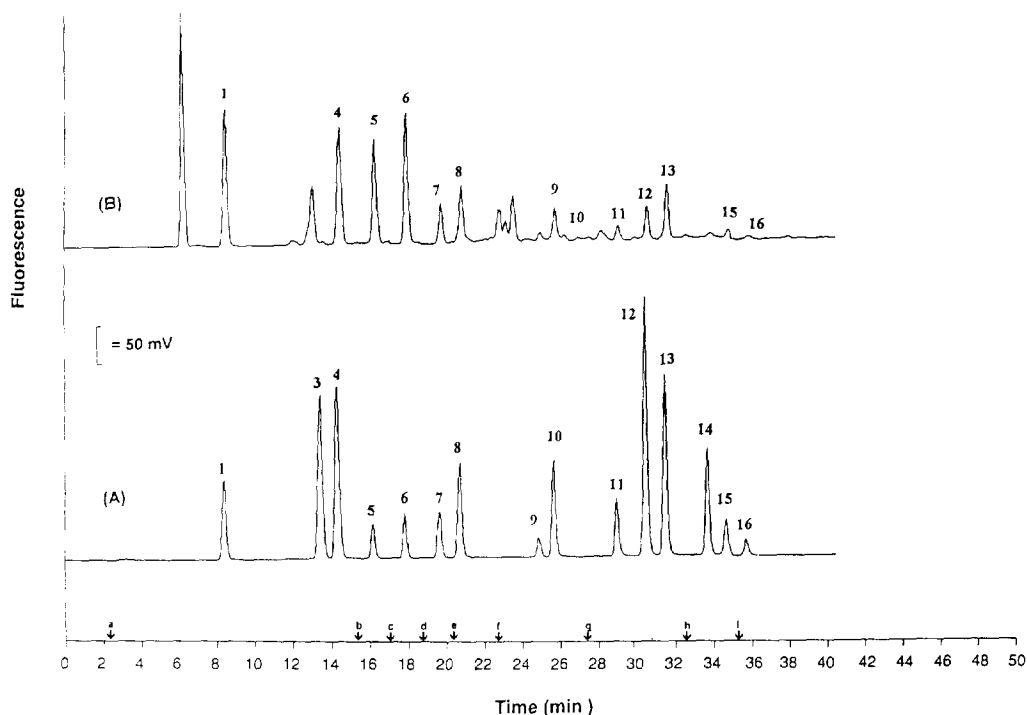


Fig. 4. Separation of (A) SRM 1647c and (B) SRM 1597, a complex mixture of PAHs from coal tar performed on Bakerbond wide-pore (300 Å) C_{18} bonded phase ($\alpha_{TBN-BaP, 20^\circ C} = 0.66$) using wavelength-programmed fluorescence detection. Conditions: mobile phase: acetonitrile–water (50:50) during 5 min, then linear gradient to 100% acetonitrile in 25 min and hold during 20 min; flow-rate: 0.5 ml/min; temperature: $20 \pm 1^\circ C$; injection of 5 μl SRM 1647c [two-fold dilution in acetonitrile–water (50:50, v/v)] and injection of 5 μl SRM 1597 [200-fold dilution in acetonitrile–water (50:50, v/v)]; fluorescence program ($\lambda_{ex}/\lambda_{em}$ in nm): a = 280/330; b = 250/370; c = 250/405; d = 280/450; e = 270/390; f = 265/380; g = 290/430; h = 290/410; i = 300/500. Peaks as in Fig. 3.

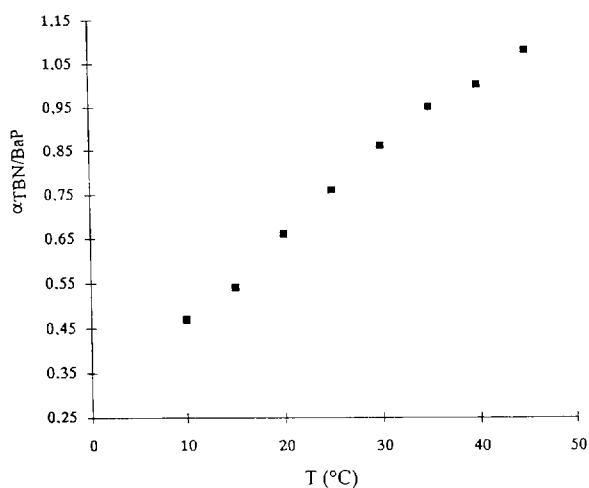


Fig. 5. Influence of the temperature on the selectivity factor $\alpha_{\text{TBN/BaP}}$. Conditions as in Fig. 2; temperatures: 10, 15, 20, 25, 30, 35, 40, 45 \pm 1°C.

at ambient and sub-ambient temperatures has been described in Ref. [20]. All six isomers were resolved with Bakerbond wide-pore C_{18} under isocratic conditions using sub-ambient temperature program. The separation demonstrates the enhancement of shape selectivity for polymeric C_{18} at lower temperatures.

The dependence of selectivity on temperature for Bakerbond wide-pore C_{18} is illustrated in a plot of $\alpha_{\text{TBN/BaP}}$ vs. temperature in Fig. 5. At elevated temperatures, the relative retention of BaP (planar shaped molecule) decreased relative to TBN (non-planar shaped molecule) which means that the $\alpha_{\text{TBN/BaP}}$ increased. At lower temperatures $\alpha_{\text{TBN/BaP}}$ decreased.

In previous studies [11,13] it was suggested that non-planar or more compact PAHs elute earlier than planar or long narrow PAHs due to differences in penetration capability into the

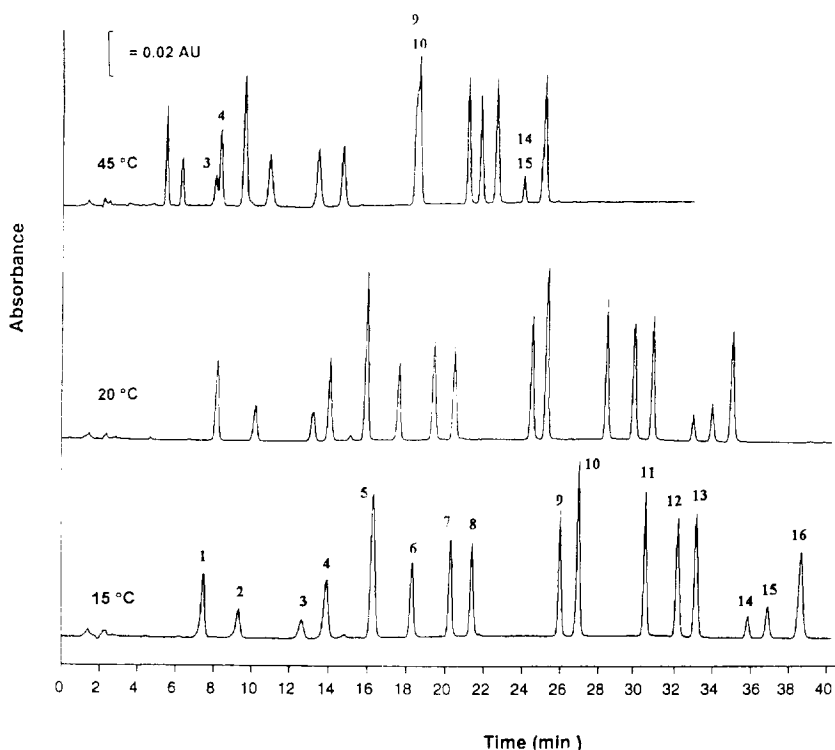


Fig. 6. Influence of the temperature on the separation of SRM 1647c. Conditions as in Fig. 3; temperatures: 15, 20, 45 \pm 1°C.

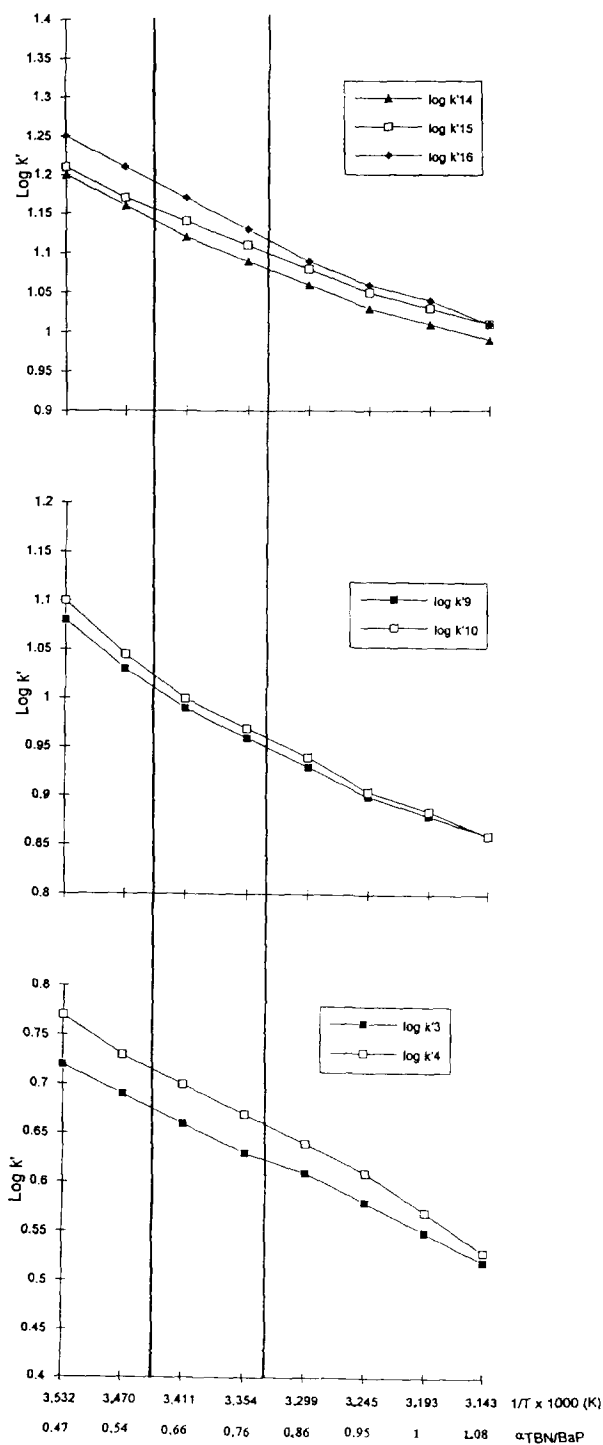


Fig. 7. Van't Hoff plots for monitoring the selectivity of some critical PAH isomers. Conditions as in Fig. 3; temperatures: 10, 15, 20, 25, 30, 35, 40, 45 \pm 1°C.

stationary phase. The results obtained here agreed with these statements.

The separation of the 16 priority pollutant PAHs is also very sensitive to temperature variations as illustrated in Fig. 6. These chromatograms illustrate clearly that the selectivity changes dramatically with the temperature.

Three Van't Hoff plots, in which the $\log k'$ of some critical PAH pairs are plotted against $1/T$, are illustrated in Fig. 7. The figure also contains temperature related $\alpha_{\text{TBN/BaP}}$ values.

The enthalpy effect ($\Delta H_{\text{m} \rightarrow \text{s}}$) of the retention process can be determined from the slope of the curve. Non-linear plots point to an existence of

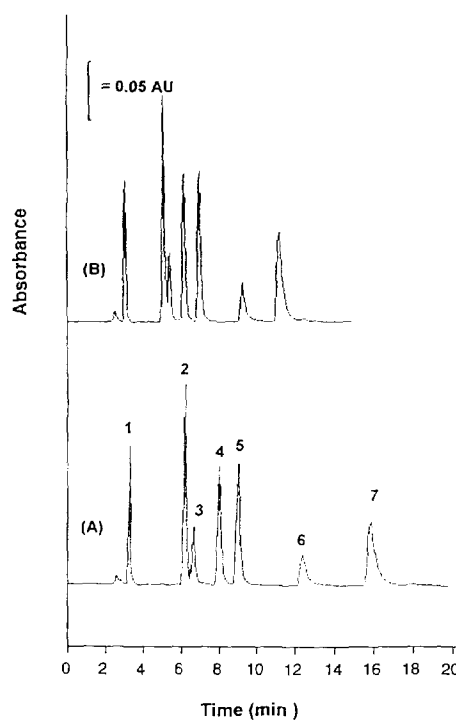


Fig. 8. Isocratic separation of seven PAHs (Borneff PAHs, including perylene) specified in DIN method 38407, Part 8, using Bakerbond wide-pore (300 Å) bonded phase. Conditions: 250 \times 3.0 mm I.D. column, 5- μ m particles; mobile phase: 100% acetonitrile at a flow-rate of 0.5 ml/min; temperature: (A) 15°C; (B) 20°C; UV detection at 254 nm; injection of 5.0 μ l PAH-7 mixture (5-fold dilution in acetonitrile). Peaks: 1 = fluoranthene (10.0 μ g/ml); 2 = benzo[b]fluoranthene (10.0 μ g/ml); 3 = perylene (5.0 μ g/ml); 4 = benzo[k]fluoranthene (10.0 μ g/ml); 5 = benzo[a]pyrene (10.0 μ g/ml); 6 = benzo[ghi]perylene (10.0 μ g/ml); 7 = indeno[1,2,3-cd]pyrene (10.0 μ g/ml).

phase transition within the bonded phase [29,30]. The presence of a transition is not observed for the wide-pore C_{18} polymeric bonded phase used in this study. Fig. 7 shows that at sub-ambient temperatures the separation of acenaphthene (peak 3) and fluorene (peak 4) is significantly enhanced (diverging curves). This is also the case for benzo[ghi]perylene (peak 15) and indeno[1,2,3-cd]pyrene (peak 16).

Two vertical lines indicate the dynamic range of the bonded phase that gives complete separation of all 16 PAHs at a given temperature and $\alpha_{TBN/BaP}$ value.

At elevated temperatures, the relative retention of dibenz[a,h]anthracene (slope of the curve is greater) is decreasing relative to benzo[ghi]perylene (peak 15); thus $\alpha_{15/14}$ is increased.

Consequently, the enthalpy effect for benzo[ghi]perylene is greater than for peak 14 and 16.

This deviating retention behaviour of ben-

zo[ghi]perylene may be due to differences in shape selectivity (benzo[ghi]perylene is also a planar shaped compound) towards the stationary phase.

The effect of the temperature on the phase selectivity is also demonstrated in Fig. 8 in which the separation of the six well known Borneff PAHs including perylene is shown. The separation between benzo[b]fluoranthene and perylene is enhanced at lower temperatures.

3.5. Effect of the gradient steepness on the separation of SRM 1647C

The influence of the slope of the gradient on the separation of SRM 1647c is illustrated in Fig. 9. Interestingly, using a steeper gradient results in a better separation of dibenz[a,h]anthracene (peak 14) and benzo[ghi]perylene (peak 15).

This selectivity enhancement is probably a result of a better solvation of the stationary

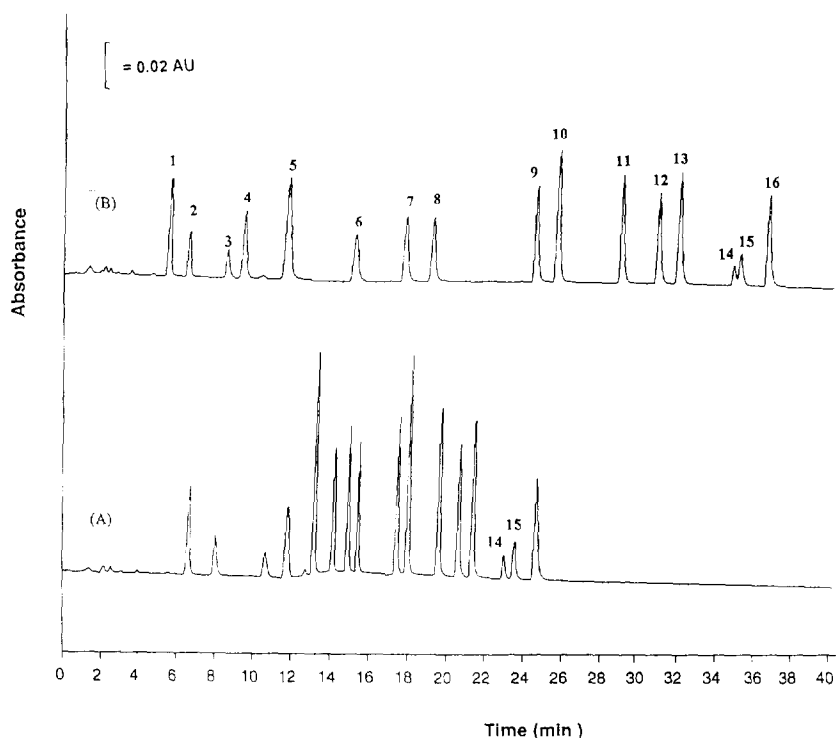


Fig. 9. Influence of the gradient steepness on the separation of SRM 1647c. Conditions as in Fig. 3. Acetonitrile–water (50:50) to 100% acetonitrile in (A) 5 min, (B) 25 min.

phase by acetonitrile compared to water/acetonitrile so that the interaction between the solute and the bonded phase layer is better. More study is necessary to proof this statement.

4. Conclusions

A novel silica based polymeric wide-pore C_{18} bonded phase is described for the separation of complex mixtures of PAHs. A selectivity test mixture is proved very useful to assess the phase selectivity. The $\alpha_{\text{TN/BaP}}$ test is routinely used by us to verify column reproducibility and assist in the adjustment of the bonding process.

Complete separation of the 16 PAHs, identified by the EPA as priority pollutants, can only be achieved using bonded phases with $\alpha_{\text{TN/BaP}}$ values in a well defined range ($0.6 < \alpha_{\text{TN/BaP}} < 0.8$).

The high selectivity of the bonded phase enables the use of fluorescence programmed-wavelength detection.

The effect of the temperature on phase selectivity is far from negligible.

The column temperature is an important parameter that can be used to modify the selectivity for PAH separations. It has been demonstrated that the selectivity parameter $\alpha_{\text{TN/BaP}}$ changes continuously with the temperature. At lower temperatures the selectivity of critical PAH isomer pairs is enhanced. The opposite trend occurs at elevated temperatures.

The slope of the gradient has a significant effects the resolution of dibenz[*a,h*]anthracene (peak 14) and benzo[*ghi*]perylene (peak 15). The separation of six Borneff PAHs inclusive perylene could be achieved under isocratic conditions using acetonitrile.

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