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Analysis and Comparison of Gas Chromatographic Columns with Polyethylene Glycol Stationary Phase

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Abstract

Gas chromatographic capillary columns with polyethylene glycol (PEG) stationary phase are very frequently used. Their polar character makes them able to analyze many molecules. Alcohols, ketones, aldehydes, and esters can be measured easily, but also molecules with acidic or alkaline characteristics can be separated. For these components, there are also acidic and alkaline modified PEG phases. When we want to buy a new column, we can choose from many manufacturers. Everyone tells us their column is the best, but we don't know the real differences between these columns. It is because every manufacturer has its testing method under different circumstances and uses different test molecules. Also, they give us only a couple of information about the column, but that is not helpful in every case.

We used an 8-component test mixture to compare the WAX columns of 6 manufacturers. The chromatogram lets us see the retention, resolution, and peak shape. We completed these parameters with the columns' efficiency and the sorption enthalpies and entropies of the analyzed compound. With these similar conditions, we can now compare the columns from different manufacturers and show more information about the columns to help the customer choose the best for the analysis.

Keywords

gas chromatography, column comparison, polyethylene glycol, efficiency, sorption enthalpy and entropy

1 Introduction

The most important part of a gas chromatographic separation is a well-chosen column with the proper stationary phase. For the analysis, we can choose columns from many manufacturers with the same dimensions and stationary phase, but they are not equivalent. The different production techniques and modifications alter possible fields of application.

Kováts [1] made the basics of the classification of the stationary phases with the introduction of the retention indices. He described these retention indices depending on the column temperature and the composition of the stationary phase. Rohrschneider [2] used five test substances (benzene, ethyl alcohol, ethyl methyl ketone, nitromethane, and pyridine) for the determination of the polarity of 20 stationary phases. He calculated the retention indices of these compounds and chose squalane as a reference apolar stationary phase. The difference in the retention indices of the compound on the examined stationary phase and squalane led him to differentiate the solutes. McReynolds [3] changed the test mixture for better accuracy. He substituted ethyl alcohol, ethyl methyl ketone, and nitromethane with 1-butanol, 2-pentanone, and nitropropane. He also suggested five more compounds (2-methyl-2-pentanol, 1-iodobutane, 2-octyn, 1,4-dioxane, and cis-hydrindane) to improve the prediction of some other kinds of molecule retention indices. He calculated the retention index difference of the ten compounds compared to squalane for 226 liquid phases and defined the average polarity of the stationary phases with the sum of the index differences of the test substances compared to squalane. Wold [4] used the McReynolds retention indices of the 226 phases with a pattern-recognition model to find similarities between the phases. 198 liquid phases were grouped into 16 clusters and 28 phases were found identical. Szentirmay et al. [5] used the ratio of the indices on the stationary phase and squalane instead of the index difference to characterize the stationary phase.

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Fernández-Sánchez et al. [6] used the Kováts' coefficient (K_c) to determine the polarity of stationary phases. The value of the coefficient increased with increasing the phenyl or the cyanopropyl groups of the 100% dimethylpolysiloxane. Castello and D'Amato [7] defined the ΔC as the difference in the carbon number of linear alkanes and alcohols with the same retention times for representing polarity differences. For capillary columns, most of these parameters are not suitable because squalane can only be used under 120 °C and the specific retention volume is unknown.

Abraham et al. [8] used the relationship between the specific retention volume and the interactions between the test solutes and 77 stationary phases. They found 3 strong acidic phases, and the remaining 74 phases were divided into 16 groups. With an improved linear solvation energy relationship coefficient model, the phase constants were recalculated for the 77 phases. With cluster analysis, these phases were divided into 16 clusters, which are consistent with Wold's [4] and their later results [9]. Poole [10] used this solvation parameter model to characterize 52 capillary columns and grouped them according to their monomer chemistry. No significant correlation was found with the McReynolds clusters, which should mean, these models define selectivity differently.

Castello et al. [11] and Vezzani et al. [12] compared gas chromatographic columns with the sorption enthalpies and entropies, based on the relationship between the retention factor and these parameters. The results correlate with the McReynolds constants, thus the columns of different manufacturers can be compared. Tudor and Oncescu [13] used *n*-alkane pairs to determine the contribution of the methylene increment to the free enthalpy ($\Delta G(CH_2)$), enthalpy ($\Delta H(CH_2)$), and entropy ($\Delta S(CH_2)$) of solution and compared them on apolar SE-30 and polar Carbowax 20M phases. Görgényi and Héberger [14] calculated the excess sorption enthalpies and entropies for alkanes, aliphatic ketones, and aldehydes and studied the effect of the column polarity on these parameters. They also determined the change in the enthalpy of the solution while the alkanes and alkanals chain length changed by a methylene group.

In our previous study [15] we compared 5% diphenyl 95% dimethyl polysiloxane columns from different manufacturers. We compared retention, peak shape, resolution, efficiency, and sorption enthalpies and entropies to find out which column for what purpose is suggested. In the literature there are no more studies about comparing the "same" stationary phase columns of different manufacturers.

In this study, we compared 11 polyethylene glycol phase capillaries from different manufacturers. We compared the chromatograms and used the solution enthalpies and entropies to find differences between these equivalent columns. Also, the efficiency was determined for the compounds on every column. With these parameters we can tell more information about the columns of the manufacturers and help the analyst by choosing with showing the differences at the same circumstances.

2 Materials, test mixture

For the column testing, we used a test mixture, which contained 8 components dissolved in methanol. The test solutes were based on the Grob test mixture but modified for the measurements [16, 17]. We needed isotherm measurements to calculate the sorption enthalpies and entropies instead the temperature-programmed Grob-test conditions. Longer carbon chain alkanes were needed to separate them from the solute and less esters for preventing co-elution. Tridecane, hexadecane, 1-decanol, 2-dodecanone, methyl dodecanoate, 2,6-dimethylphenol, 2,6-dimethylaniline and *N*,*N*-dicyclohexylamine were purchased from Sigma Aldrich. 20–30 mg of each compound were added into a 25 ml volumetric flask and diluted with methanol. 5 ml of the stock solution was diluted to 100 ml in a volumetric flask and the final concentrations of each component were about 50 µg ml^{-1}. The mixture was held at 4 °C.

3 Methods, tested columns

We made our measurements with a Shimadzu GC-2014 gas chromatograph with an AOC-20i+s autosampler. The injector was held at 200 °C, the carrier gas was hydrogen, and the split ratio was 20. For the efficiency measurements, the column temperature was held at isotherm 140 °C and the linear velocity was 10, 20, 30, 40, 50, 75, 100, and 150 cm s^{-1}. For the determination of the sorption enthalpies, the linear velocity was 50 cm s^{-1} and the column temperature was isotherm 80, 100, 120, 140, 160, 180, and 200 °C. The flame ionization detector was held at 220 °C.

The examined columns were classified into two groups based on column dimensions. We tested 4 columns with 30 m length, 0.32 mm inner diameter, and 0.5 µm film thickness: CP-WAX 52CB from Chrompack, two ZB-WAX (marked as ZB-WAXa and ZB-WAXb) from Phenomenex, and Solgel-WAX from Supelco. With 30 m length, 0.25 mm inner diameter, and 0.25 µm film thickness, we measured 8 columns: DB-WAX, DB-WAX UI, and VF-WAXms from Agilent, ZB-WAX and ZB-WAX Plus from Phenomenex, MEGA-WAXms from MEGA, Stabilwax from Restek and Supelcowax-10 from Supelco. The Stabilwax and Supelcowax-10 columns were old and were used for many measurements, but it was important to know, which can be used for more analysis.

For the determination of the efficiency of the columns we used and the number of theoretical plates (*N*) and the height equivalent to a theoretical plate (*HETP*). We used for the calculations Eqs. (1)–(3),

$$\omega = \frac{A}{h} \tag{1}$$

$$N = 5.56 \times \left(\frac{t_R}{\omega}\right)^2 \tag{2}$$

$$HETP = \frac{L}{N} \tag{3}$$

where ω is the peak width at half height, A is the area under the peaks and h is the height of the peaks, t_R is the retention time and L is the length of the column.

Then we plotted the *HETP* values as a function of the linear velocity (*H-u* chart) for every compound on every column and the minimum of the curves represents the maximum of efficiency.

To calculate the sorption enthalpies and entropies we determined the phase ratio (β) first with Eq. (4),

$$\beta = \frac{V_m}{V_s} \tag{4}$$

where V_m is the volume of the mobile phase and V_s is the volume of the stationary phase.

We used the retention time of the compound (t_R) and methane (unretained compound) (t_0) and the phase ratio (β) to calculate the partition coefficient (K) with Eq. (5).

$$K = \frac{t_R - t_0}{t_0} \times \beta \tag{5}$$

The free enthalpy of solution (ΔG) equals the logarithm of the partition coefficient multiplied with the temperature in Kelvin (T) and the universal gas constant (R) and it is also the difference of the enthalpy of solution (ΔH) and the entropy of solution (ΔS) multiplied with the temperature like shown in Eq. (6).

$$\Delta G = R \times T \times \ln K = \Delta H - T \times \Delta S \tag{6}$$

We plotted the logarithm of the partition coefficient as a function of the reciprocal of temperature, like Eq. (7).

$$\ln K = \frac{\Delta H}{R \times T} - \frac{\Delta S}{T} \tag{7}$$

We fit straight on the points and the R^2 value was over 0.995. Then we calculated the enthalpies of solution from the slope (a) with Eq. (8) and sorption entropies from the intercept (b) with Eq. (9). We wanted to calculate the enthalpies of solution from the viewpoint of the solution, so we needed to take this value with a negative sign.

$$\Delta H = R \times a \tag{8}$$

$$\Delta S = -R \times b \tag{9}$$

With these considerations the less enthalpy of solution shows stronger interactions between the molecule and the stationary phase.

4 Results and discussions

4.1 30 m × 0.32 mm × 0.5 µm columns

The chromatograms of the 4 columns are shown in Fig. 1. These chromatograms have many differences at the standard measurement conditions with 50 cm s^{-1} linear velocity and 140 °C oven temperature. The two ZB-WAX columns have similar patterns. The Solgel-WAX column has the shortest retention times, while the CP-WAX 52 CB column has the highest retention. This means, CP-WAX 52 CB is good for the separation of complex samples, but Solgel-WAX should be used for samples only with a couple of compounds at the same temperature. On the CP-WAX 52 CB column, the third component, the *N*,*N*-dicyclohexylamine has a broad peak, which means it has stronger interactions with the stationary phase. A slight peak tailing can be

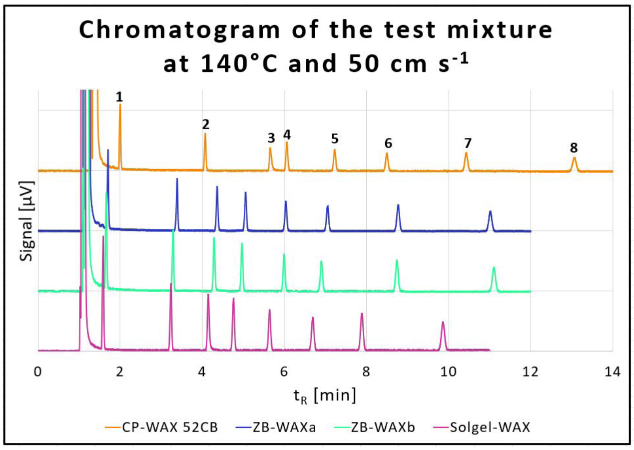

Fig. 1 The chromatogram of the test mixture at 140 °C and 50 cm s^{-1} on the 30 m × 0.32 mm × 0.5 µm columns 1 – tridecane, 2 – hexadecane, 3 – *N*,*N*-dicyclohexylamine, 4 – 2-dodecanone, 5 – 1-decanol, 6 – methyl dodecanoate, 7 – 2,6-dimethylaniline, 8 – 2,6-dimethylphenol

noticed for the *N*,*N*-dicyclohexylamine on the Solgel-WAX column too. These peak shapes are shown on Fig. 2. This peak broadening and strong interaction cause the worse, but still enough resolution on the CP-WAX 52 CB column. On the other columns, the resolution is similar.

The *H*-*u* charts are similar for every compound. The *H*-*u* charts of *N*,*N*-dicyclohexylamine are shown in Fig. 3. The CP-WAX 52 CP column has the best efficiency, and the ZB-WAX columns are the second. The Solgel-WAX with its low retention has the highest *HETP* values. The tailing of the *N*,*N*-dicyclohexylamine on the CP-WAX 52 CB column causes higher *HETP* values, but the peak broadening is correlated with the higher retention time. The efficiency is slightly worse for this component than the two ZB-WAX columns.

We calculated the sorption enthalpies and entropies for each component. These results are shown in Table 1. The sorption enthalpies are similar in most cases. The only significant difference is by *N*,*N*-dicyclohexylamine. The enthalpy is much lower on the CP-WAX 52-CB column, which means the stationary phase has stronger interactions with basic compounds. So, this column's stationary phase has an acidic character, even if it is not nitroterephtalic acid modified polyethylene glycol. The Solgel-WAX column has also lower values than the ZB-WAX columns, but in this case, the interactions are not too strong. These results are consistent with the peak shape on the chromatograms. The entropies are similar and three magnitudes lower than the enthalpies. It means the enthalpy has a significant effect on the separation.

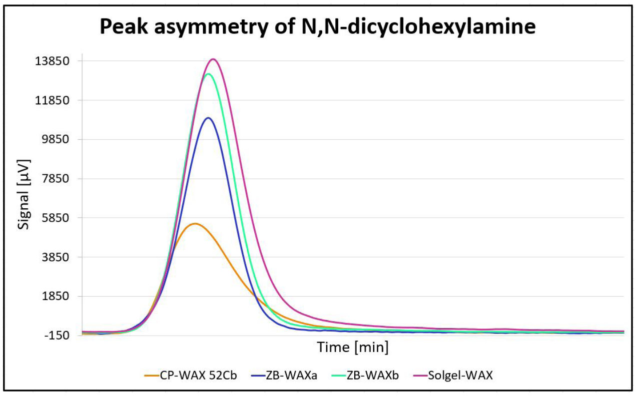

Fig. 2 The peak shape of *N*,*N*-dicyclohexylamine at 140 °C and 50 cm s^{-1} on the 30 m × 0.32 mm × 0.5 µm columns

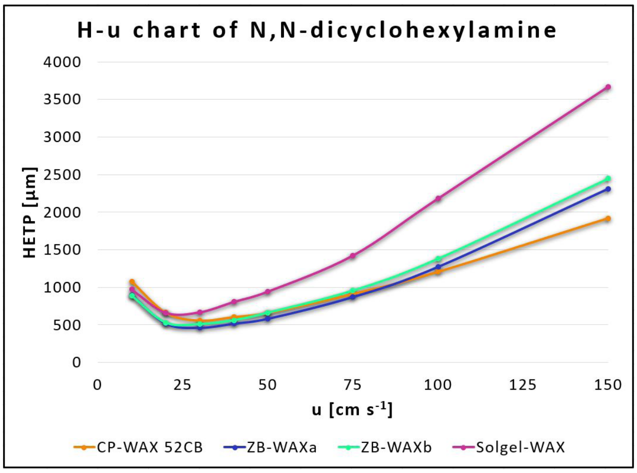

Fig. 3 The *H*-*u* charts of *N*,*N*-dicyclohexylamine at 140 °C

These results help us to choose the best column for our analysis. The CP-WAX 52 CB column has high retention times, which is good for the separation of compounds with similar retention indices. Even with high retention, the peaks are narrow, and it results in good efficiency. The stationary phase has an acidic character, so analysis of basics is not recommended on this column. The two ZB-WAX columns have the same results. They have good retention and symmetric peaks for every analyte. The efficiency is also good, and the interactions are the weakest for most of the compounds. This means we can use these columns in many cases for complex samples, even for basic components. The Solgel-WAX column has the lowest retention, so this could be used for samples with a few components with different retention indices. The low retention causes worse efficiency than the other columns, but the peaks are symmetric. The stationary phase is slightly acidic, but we can measure basic compounds on it.

4.2 30 m × 0.25 mm × 0.25 µm columns

The chromatograms of the test mixtures are shown in Fig. 4. By these columns, we found many differences. The two heavily used columns have many differences. The Supelcowax-10 column has broad and short peaks for every component. During its use, its stationary phase broke down and now it is not able to analyze anything. Although the Stabilwax column was used also for a long time (produced in 1993), it could be used for more measurements. It shows us the importance of column testing from time to time. Even if Stabilwax is not appropriate for its original use, it has potential for the analysis of other kind of compounds. The Supelcowax-10 has a broken stationary phase, and it cannot be used for any measurement, so we don't show more of its poor results.

The chromatograms of the remaining 7 columns have many different patterns. The retention is low on Stabilwax and ZB-WAX column, medium on MEGA-WAXms, ZB-WAX Plus, and VF-WAXms, and higher on DB-WAX and DB-WAX UI. At 140 °C and 50 cm s^{-1}, we found 2.3 minutes difference in the retention time of the last

Table 1 The calculated sorption enthalpies and entropies for each component on the 30 m × 0.32 mm × 0.5 µm columns

	n-tridecane		*n*-hexadecane		*N*,*N*-dicyclohexylamine		2-dodecanone	
	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]
CP-WAX 52 CB	−38.3	54.4	−49.3	70.0	−52.9	74.4	−51.1	69.8
ZB-WAXa	−36.8	53.1	−48.7	69.5	−45.9	59.6	−50.8	69.8
ZB-WAXb	−37.6	50.6	−48.9	68.6	−46.1	59.0	−50.9	69.4
Solgel-WAX	−37.5	52.4	−48.9	68.8	−47.3	62.1	−50.8	69.3
	1-decanol		methyl dodecanoate		2,6-dimethylaniline		2,6-dimethylphenol	
	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]
CP-WAX 52 CB	−55.3	78.3	−55.2	76.5	−53.3	69.9	−57.7	78.3
ZB-WAXa	−55.1	78.3	−55.0	76.4	−53.3	70.1	−57.7	78.8
ZB-WAXb	−55.3	77.8	−55.0	76.1	−53.4	69.9	−57.9	78.4
Solgel-WAX	−55.1	77.9	−54.9	75.7	−53.0	69.6	−57.5	78.1

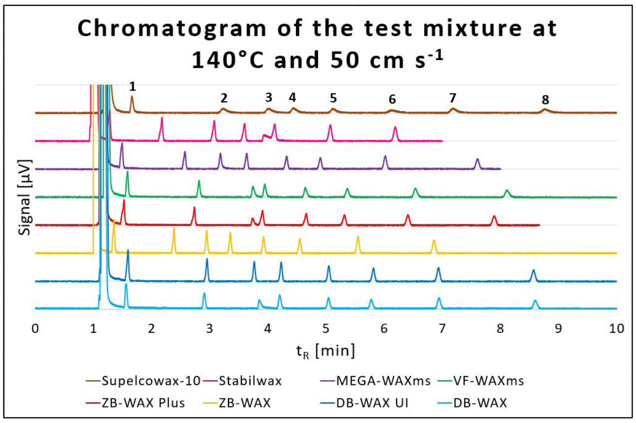

Fig. 4 The chromatogram of the test mixture at 140 °C and 50 cm s^{-1} on the 30 m × 0.25 mm × 0.25 µm columns 1 – tridecane, 2 – hexadecane, 3 – *N*,*N*-dicyclohexylamine, 4 – 2-dodecanone, 5 – 1-decanol, 6 – methyl dodecanoate, 7 – 2,6-dimethylaniline, 8 – 2,6-dimethylphenol

component, while it elutes before 9 minutes. The third peak, the *N*,*N*-dicyclohexylamine has broad peaks on many columns. The best peak shape for this compound is on the ZB-WAX column. But on the Stabilwax column, its peak position changed, it eluated as the fifth peak. This retention order is changed at other oven temperatures too, which is shown in Fig. 5. This change did not appear on the other columns. This could be caused by the acidity of the column. The distorted *N*,*N*-dicyclohexylamine peak and the retention have a huge factor on the resolution of the third and the fourth peaks. The peak shapes of *N*,*N*-dicyclohexylamine is shown in Fig. 6. The DB-WAX, DB-WAX UI, and MEGA-WAX could separate these components with a broad amine peak, but VF-WAXms and ZB-WAX Plus could not separate them. On ZB-WAX the peak is symmetric and these components are separated well.

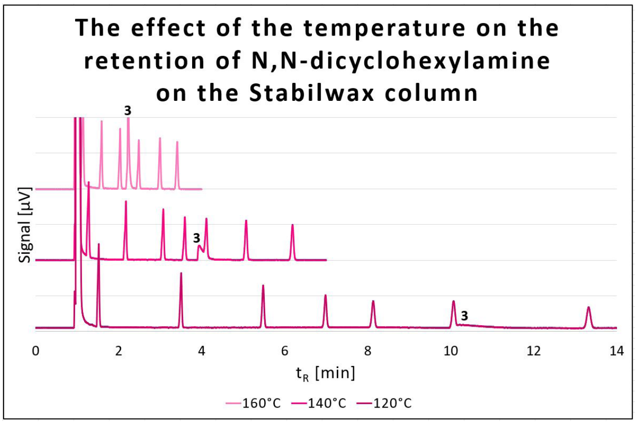

Fig. 5 The effect of the temperature on the retention of *N*,*N*-dicyclohexylamine (3) on the Stabilwax column

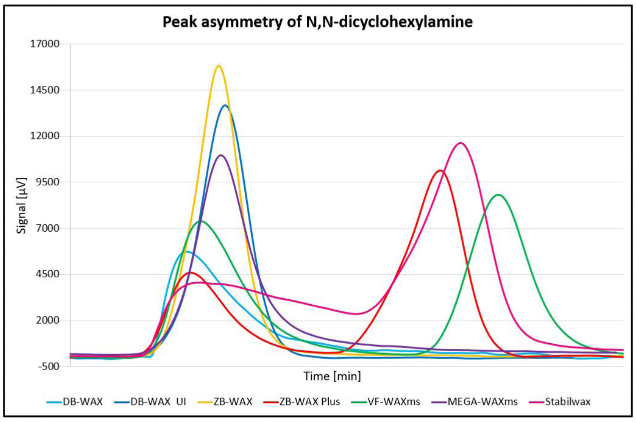

Fig. 6 The peak shape of *N*,*N*-dicyclohexylamine at 140 °C and 50 cm s^{-1} on the 30 m × 0.25 mm × 0.25 µm columns

The *H*-*u* charts of the columns are shown in Fig. 7. The DB-WAX UI has the best efficiency by the optimum. At increased velocities, its efficiency deteriorates barely. The VF-WAXms column has higher *HETP* values at low

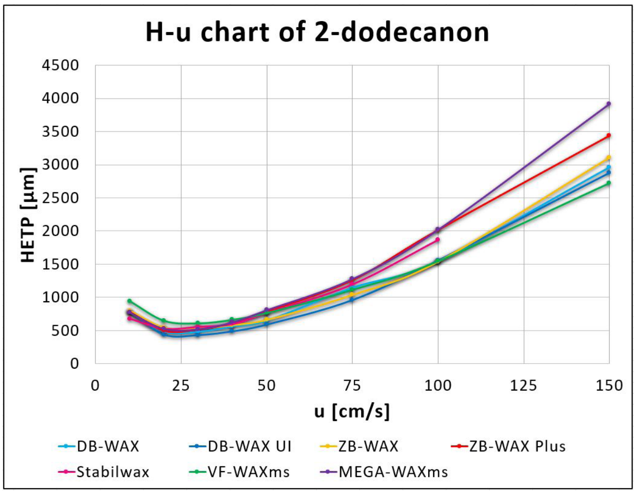

Fig. 7 *H-u* chart of 2-dodecanone at 140 °C

linear velocities, but it slightly increases at higher linear velocities. The ZB-WAX, ZB-WAX Plus, and DB-WAX have good efficiency, but at high velocities, their *HETP* value increases significantly. The MEGA-WAXms column has worse efficiency for components with low retention, but it is much better at higher retention times. The Stabilwax column has extremely high *HETP* values for the *N*,*N*-dicyclohexylamine, but for the other compounds, it has similar efficiency, to the other columns.

The sorption enthalpies are shown in Table 2. The ZB-WAX Plus column's stationary phase has the weakest interactions with almost every kind of molecule. The MEGA-WAXms has the weakest interactions with the amine compound. The sorption enthalpies are similar on every column, except for the *N*,*N*-dicyclohexylamine. On the Stabilwax column, it has a very low enthalpy, which means it has very strong interactions with the amines. This has a very acidic stationary phase, which could occur when the polyethylene glycol stationary phase got old. VF-WAXms has also lower enthalpies and strong interactions, but not too strong to change the retention order. The other columns have no significant difference in this value. These differences can be seen on the chromatograms when we analyze the *N*,*N*-dicyclohexylamine peak shape.

Our results show the importance of column testing. The old, used a lot of columns are not always useless. The Supelcowax-10 column is not suitable for any measurement. But maybe the old Stabilwax column is not good for the original analysis, it could be used for the analysis of many components. Its stationary phase is very acidic, it has a problem only with basic components. In other cases, it has good efficiency, and peak shape, but its retention is low. It can be used for simple matrix samples, just like ZB-WAX. ZB-WAX also has the best peak shape for every component, and its efficiency is good. It could be well used for any kind of molecule. ZB-WAX Plus, VF-WAXms, and MEGA-WAXms have medium retention and are able for better separation. These columns have good efficiency and ZB-WAX Plus has the weakest

Table 2 The calculated sorption enthalpies and entropies for each component on the 30 m × 0.25 mm × 0.25 μm columns

	n-tridecane		*n*-hexadecane		*N*,*N*-dicyclohexylamine		2-dodecanone	
	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]
DB-WAX	−37.4	44.6	−49.0	61.9	−47.6	54.9	−50.7	61.7
DB-WAX UI	−36.3	41.4	−48.8	61.1	−45.0	49.1	−50.5	61.1
ZB-WAX	−38.1	47.6	−49.0	62.9	−44.5	49.8	−50.7	62.9
ZB-WAX Plus	−33.8	36.1	−46.8	57.2	−46.3	52.4	−49.0	58.1
VF-WAXms	−37.2	45.4	−49.1	63.3	−48.7	58.7	−52.6	67.3
MEGA-WAXms	−37.7	47.1	−48.4	62.1	−43.5	47.6	−50.3	62.4
Stabilwax	−37.4	46.3	−48.4	62.2	−60.9	84.8	−49.7	60.8
	1-decanol		methyl dodecanoate		2,6-dimethylaniline		2,6-dimethylphenol	
	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthalpy [kJ mol^{-1}]	Entropy [J K^{-1}]
DB-WAX	−55.4	70.8	−54.9	68.3	−52.9	61.8	−57.4	70.4
DB-WAX UI	−55.2	70.4	−54.8	68.2	−52.9	61.7	−57.3	70.3
ZB-WAX	−54.9	71.4	−54.9	69.8	−53.2	63.5	−57.5	72.0
ZB-WAX Plus	−53.5	67.1	−53.3	65.2	−51.5	59.0	−55.8	67.4
VF-WAXms	−55.1	71.6	−55.0	69.9	−53.1	63.4	−57.5	71.9
MEGA-WAXms	−54.8	71.3	−54.6	69.3	−53.0	63.4	−57.7	72.4
Stabilwax	−55.2	72.5	−54.5	69.2	−52.9	63.3	−57.2	71.7

interactions with almost every compound. Only the amine has stronger interaction with the stationary phase. For amines, the MEGA-WAXms, ZB-WAX, and DB-WAX UI are the best choice. DB-WAX UI and DB-WAX have the highest retention times, and DB-WAX UI has the best efficiency by the optimal linear velocity. The DB-WAX is not enough inert to analyze basics with it.

5 Conclusion

The WAX columns with polyethylene glycol stationary phase are commonly used for measurements of polar molecules. But when we choose the column, we should know some information about them to use the best for our analysis. The manufacturers give us some data, but it is better if we know much more about our columns.

We used an eight-component test mixture to see the differences between the chromatograms under similar circumstances, to investigate the retention pattern and efficiency of the columns, and to calculate the sorption enthalpies and entropies. These parameters let us select the best column if we know the sample's complexity and the characteristic of the analyzed components. The main difference between the columns from different manufacturers is in the acidity of the stationary phase, even if they weren't acidic modified polyethylene glycol phases.

By many companies, the columns are used only for one type of analysis and when they start to underperform, these columns are thrown away. But after a simple testing, there is a chance to use the column for another kind of analysis.

With a couple measurements we are able to get more information than the manufacturers tell us. The sorption enthalpies and entropies let us know the strength of the interaction between the stationary phase and the different kind of molecules. The *H-u* charts show more about the efficiency than just one *HETP* value for a compound. This helps the analyst to get the best results with the well-chosen column. Also, the lifetime of these columns can be increased, if we can use them for other measurements after it is not appropriate for the original task.

Acknowledgment

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