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Investigation and Comparison of 5 % Diphenyl – 95 % Dimethyl Polysiloxane Capillary Columns

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Abstract

In gas chromatography, for the analysis, the most important thing is the well-chosen column. After having decided about which stationary phase is the best for our measurement and we know the length, the inner diameter and film thickness, we can buy the column from a lot of manufacturers. The products of these manufacturers' look similar to each other, but they are different, because of the different manufacturing technologies. These differences could have significant influence on the separation. It is so important to compare the „same" columns, despite the manufacturers' efforts to produce the best quality columns.

5 % diphenyl – 95 % dimethyl polysiloxane stationary phase is widely used. Its slight polarity makes it able to determine very different compounds from the alkanes, through volatiles, drugs, fatty acid methyl esters, amines or phenols.

In our work, we tested 5 % diphenyl – 95 % dimethyl polysiloxane stationary phase columns with an 8-component mixture. These columns were from different manufacturers. During isothermal conditions, we determined the height equivalent of theoretical plates on 8 linear velocity level. We represented these parameters as the function of linear velocity. With a constant linear velocity, we measured the excess sorption enthalpy and entropy. From the chromatograms we spotted differences in the retention and in resolution, which is important if we use the column for volatiles or for complex samples. With these parameters we can show many differences between the columns. These parameters have significant influence when we want to use our column for a given task.

Keywords

gas-chromatography, column testing, diphenyl-dimethyl polysiloxane, efficiency, excess enthalpy and entropy

1 Introduction

In chromatography, one of the biggest problems is to predict the retention of the molecules on the proper stationary phase. It is more difficult, if we have to decide, which column to buy. There are many manufacturers and they have similar columns. When we want to adapt a method, only the recommended stationary phase is determined. The manufacturers use different technologies when they create their product and it results in different characteristics for the stationary phase. They try to persuade us why their column is the best for the analysis, but very often it becomes harder for the analyst to choose.

The first attempt to describe the elution was made by Ervin Kovats [1]. He used the observation that the reduced retention time of homologous series of any kind of molecules is exponentially growing with the carbon number. He defined the retention index of n-alkanes as one hundred times of their carbon number. This led him to a scale, where any other molecule retention index can be calculated from the reduced retention times of the adjacent n-alkanes and the compound. This index is constant at similar circumstances [1-3].

Rohrschneider [4] used these retention indices andfive standard molecules (benzene, ethanol, 2-butanone, nitromethane and pyridine) to determine the polarity of the stationary phase, compared to squalane. These reference standards show the different intermolecular interactions with the stationary phase [2-4]. McReynolds [5] changed ethanol to n-butanol, 2-butanone to 2-pentanone and nitromethane to nitropropane. They compared the difference of the retention indices of

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these compounds on squalane and the involved stationary phase, which led them to the polarity scale of the columns. The Chromatographic Polarity (CP) can be calculated, as shown in Eq. (1),

$$CP = \frac{\sum_{i=1}^{5} \left(\Delta I\right)_i^{F}}{\sum_{i=1}^{5} \left(\Delta I\right)_i^{OV-275}} \times 100, \qquad (1)$$

where $\left(\Delta I\right)_i^F = I_i^F - I_i^{sq}$ is the difference in the indices of the i-th model material on the given stationary phase (*F*) and squalane (*sq*), while $\left(\Delta I\right)_i^{OV-275} = I_i^{OV-275} - I_i^{sq}$ [2, 3, 5]. The main problem is the necessity of measurements on three different columns and the analysis on squalane is possible up to 100 °C.

In the 90s Abraham et al. [6, 7], and Abraham and Whiting [8] tried to improve McReynolds' model. They built up a model between the specific retention volume and the solvation interactions of these molecules. With this model, they characterized many stationary phases and determined the solvation parameters of alkylaromatic hydrocarbons [6-8].

In 2003, Szepesy analyzed 19 different reverse phase HPLC columns, using the linear solvation energy model. He determined the different selectivity factors and the effect of the mobile phase on these factors [9]. Larbi et al. used the retention indices and the linear solvation energy of monosubstituated benzenes to characterize eight RPLC columns. They showed that these parameters are good for modelling molecular interactions and retention mechanism, using different stationary phases [10].

In supercritical fluid chromatography West et al. analyzed columns. They used the linear solvation energy model and tried to indicate the differences with their sum of ranking differences model. They selected one reference column and compared 70 other to it with the differences in the retention of 86 standard compounds. This model had similar results as the linear solvation energy model and the sum of ranking differences model is able to determine the column differences [11]. Desfontaine et al. tested three new SFC stationary phases with basic compounds. They analyzed the peak shapes and the effect of ammonium formate eluent modifier. They found one of the new columns is not suitable for basic compounds. The other two was compared to two well-known stationary phases and the retention times of the compound led them to choose the appropriate column [12].

In gas chromatography, D'Archivio and Giannitto used the quantitative structure-retention relationship approach with multilinear regression model. With theoretical molecular descriptors they were able to determine the retention and distinguish the columns [13]. Poole tested 52 different columns and determined the McReynolds phase constants on different temperatures. He tried also some stationary phases, which structure is not described by manufacturer [14].

The classification of the gas chromatographic columns was earlier described by many models, but there are only few articles on this topic. Nowadays there are some examples in HPLC, SFC and GC to characterize these columns. But this happens with different type of stationary phases or column dimensions. We decided to compare similar phases with equal column sizes made by different manufacturers. We used more parameters, such as efficiency, solvation excess enthalpy and entropy, resolution, peak shape or retention to find the differences and similarities. These led us to provide more information about the columns' substitutability in the daily analytical process.

2 Materials, test mixtures

We selected 8 different test molecules that can represent the main interactions with the stationary phases. Tridecane and hexadecane for dispersion interaction, 1-decanol and methyl dodecanoate as electron pair acceptor, 2-dodecanone as electron pair donor, 2,6-dimethylphenol as acidic compound and 2,6-dimethylaniline and *N*,*N*-dicyclohexylamine as bases. The standards were purchased from Sigma-Aldrich. 50-75 mg of the components were added into a 50 ml metric flask and were diluted with methanol. 2.5 ml of this solution was diluted to 50 ml, so the final concentration was 50-75 μg ml-1 for each analyte.

3 Methods

We used a SHIMADZU-2014 gas chromatograph with an AOC-5000 autosampler. 1 μl sample was injected to the GC. The injector was thermostated at 200 °C. We used hydrogen as carrier gas and split ratio was 20. For the thermodynamic analysis, linear velocity was always 50 cm s-1. For the efficiency – linear velocity function, we used 10, 20, 30, 40, 50, 75, 100 and 150 cm s-1 linear velocities. We used isotherm column temperature conditions for the analysis. By the efficiency measurements the oven temperature was 140 °C. Thermodynamic methods were at isotherm 80 °C, 100 °C, 120 °C, 140 °C, 160 °C, 180 °C and 200 °C oven temperature measured. The flame ionization detector was held at 220 °C. We used GC Solution software for data analysis. The three 30 m × 0.25 mm × 0.25 μm columns were Rtx-5 from Restek, ZB-5 from Phenomenex and BPX-5 from Supelco. The eight 30 m × 0.32 mm × 0.25 µm columns were four Rtx-5 from Restek, one ZB-5HT Inferno from Phenomenex, one BPX-5 from Supelco and two HP-5 from Agilent. In cases when we had more columns with the same name and dimensions, we used letters (a, b, c or d) in this article to distinguish them. The measurements were repeated five times at each temperature and linear velocity.

By the thermodynamic analysis, we only needed the retention times of each component on different temperature, the dimensions of the column, and the retention time (t_R) of methane (unretained molecule) (t_0). Then we calculated the phase ratio (β) with Eq. (2) and the partition coefficient (K) with Eq. (3),

$$\beta = \frac{V_m}{V_s}, \tag{2}$$

$$K = \frac{t_R - t_0}{t_0} \times \beta, \tag{3}$$

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

The logarithm of partition coefficient multiplied with the temperature (T) and the universal gas constant (R), is the free enthalpy (ΔG), like shown in Eq. (4). We made a graph from the logarithm of the partition coefficients as a function of reciprocal of temperature (T) in Kelvin, like Eq. (5). We fit straight on these points, and the R^2 values were above 0.995.

$$\Delta G = RT \ln\left(K\right) = \Delta H - T\Delta S \tag{4}$$

$$\ln\left(K\right) = \frac{\Delta H}{R} \times \frac{1}{T} - \frac{\Delta S}{R} \tag{5}$$

We calculated the enthalpy (ΔH) from the slope (a) of the curve with Eq. (6) and the entropy (ΔS) from the intercept with Eq. (7). We want to calculate these values from the view solution, so we needed to take this value with negative sign. By this modification, the less excess enthalpy (ΔH_e) shows stronger interactions.

$$\Delta H_s = -\frac{a}{R} \tag{6}$$

$$\Delta S = -\frac{b}{R} \tag{7}$$

For the excess enthalpies, we looked up the molar vapor enthalpies (ΔH_v) of each component in databases. The excess enthalpy is the difference of the calculated sorption enthalpy and the molar vapor enthalpy like in Eq. (8).

$$\Delta H_e = \Delta H_s - \Delta H_v \tag{8}$$

After that, we compared the enthalpy and entropy values to know which parameter has the significant effect on the separation.

4 Results and discussion

4.1 30 m × 0.32 mm × 0.25 µm columns

The chromatograms of these columns are shown in Fig. 1. In this case we haven't found any important differences, only ZB-5HT Inferno has higher retention for the test components. At different temperatures and linear velocity, the pattern is the same. But when we analyzed the efficiency as a function of linear velocity (H-u chart), we could find some more valuable differences. HP-5b and Rtx-5b has a bit higher H-values for each component. But ZB-5HT Inferno with its higher retention has much better efficiency than the others. This longer retention let us separate better complex matrices or just simply better resolution. The critical pair for separation is the third and fourth peak, 1-decanol and n-tridecane. At higher temperatures or linear velocities, Rtx-5a and Rtx-5b could not separate these components. The H-u chart for 2,6-dimethylaniline is shown on Fig. 2.

From the measurements at different isotherm temperatures the thermodynamic parameters of these columns were calculated, such as excess enthalpy and excess entropy. The calculated excess enthalpies and entropies are shown in Table 1. In many cases these values are similar. But BPX-5 has lower excess enthalpy for all the measured molecules. By every compound, HP-5a and ZB-5HT Inferno has a bit lower values. This means, these columns establish stronger interactions with the analytes, than the others. The big difference is in the case of *N,N*-dicyclohexylamine, where the values are very low.

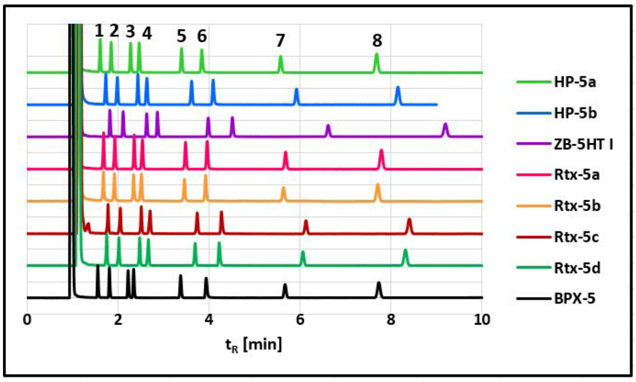

Fig. 1 Chromatogram of the text mixture on the investigated columns at 140 °C and 50 cm s-1. 1 – 2,6-dimethylphenol, 2 – 2,6-dimethylaniline, 3 – 1-decanol, 4 – n-tridecane, 5 – 2-dodecanone, 6 – *N,N*-dicyclohexylamine, 7 – methyl dodecanoate, 8 – n-hexadecane

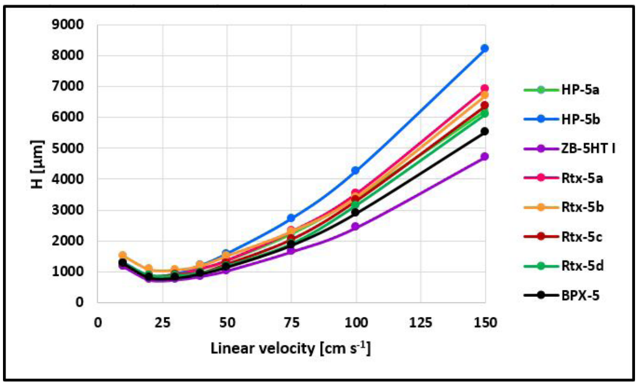

Fig. 2 The H-u chart of 2,6-dimethylaniline on 140 °C

It means, these columns' surface is acidic and has a strong interaction with basic components. This is not too strong, because the 2,6-dimethylaniline has normal excess enthalpy values. Except BPX-5, where the stationary phase has strong interactions with aniline and other components and more acidic than any other column. Entropies are the same and three magnitudes lower than enthalpies for every column and component. This means, the enthalpy controls the process of the separation in every case.

These results show that the chromatogram from the manufacturer does not give us enough information about the column. The H-u charts and the excess enthalpies tell us much more. The chromatograms look similar, but ZB-5HT Inferno has better efficiency and a bit longer retention, which allows the separation of complex matrices. HP-5a and Rtx-5b have higher values than the others. The resolution of peak #3 and #4 is not enough in many cases, so these should not be used for measurement of molecules with close retention indices. From the thermodynamic data we received information about the interactions between the sample molecules and the stationary phase. By every component, BPX-5 had stronger interactions, so it should be used only for slightly polar and non-polar compounds. HP-5a, ZB-5HT Inferno and BPX-5 has lower excess enthalpy values for *N*,*N*-dicyclohexylamine, so the stationary phase is acidic and strong acid-base interaction occurs with basic molecules. It means, these columns should be used for determination of acidic or neutral compounds.

4.2 30 m × 0.25 mm × 0.25 µm columns

The chromatograms of the columns are shown in Fig. 3. In this case, there are many differences with the default settings (Fig. 3. above), the 6th peak on BPX-5 is hardly noticeable and at lower temperature, the peak disappeared. Evaluable peak was only detected when the linear velocity (Fig. 3. middle) or the temperature (Fig. 3. below) was increased. ZB-5 has also a wide peak for *N*,*N*-dicyclohexylamine, but it can be integrated. This means the interactions are much stronger then Rtx-5. BPX-5 also has peak broadening for the other polar compounds. It confirmed that H-bond can be formed with the stationary phase. Rtx-5 has tailing only for 1-decanol and *N*,*N*-dicyclohexylamine. The resolution for 1-decanol and n-tridecane is also an important difference. BPX-5 has low retention and it causes, that these peaks cannot be separated. ZB-5 and Rtx-5 can separate them at a lower temperature or linear velocity, but ZB-5 has better resolution. The efficiency for ZB-5 and Rtx-5 by low linear velocity is similar.

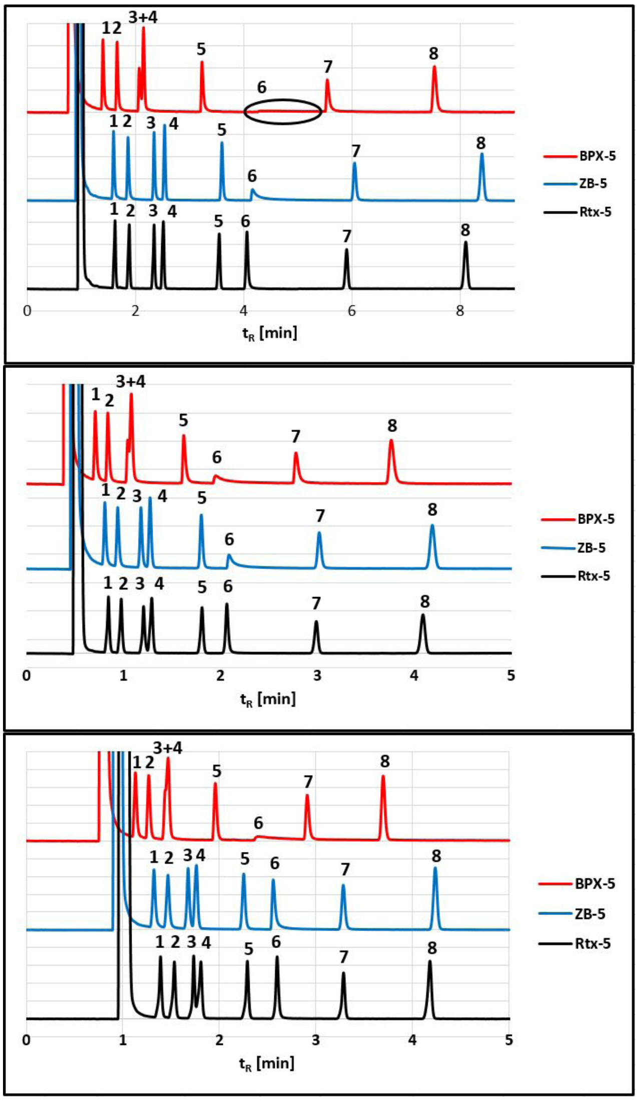

Fig. 3 Chromatogram of the text mixture on the investigated columns at 140 °C and 50 cm s-1 (above), 140 °C and 100 cm s-1 (in the middle) and 160 °C and 50 cm s-1 (below).

1 – 2,6-dimethylphenol, 2 – 2,6-dimethylaniline, 3 – 1-decanol, 4 – n-tridecane, 5 – 2-dodecanone, 6 – *N*,*N*-dicyclohexylamine, 7 – methyl dodecanoate, 8 – n-hexadecane

Table 1 Calculated excess enthalpies and entropies for each component on the 30 m × 0.32 mm × 0.25 µm

	2,6-dimethylphenol		2,6-dimethylaniline		1-decanol		n-tridecane		2-dodecanone		*N,N*-dicyclohexylamine		Methyl dodecanoate		n-hexadecane	
	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]
HP-5a	35.4	46.7	17.8	48.7	29.6	61.4	16.0	65.0	18.2	68.3	3.1	60.6	17.5	77.0	19.0	80.8
HP-5b	36.0	45.4	18.2	47.7	30.1	60.4	16.4	64.2	18.6	67.7	3.4	60.1	17.8	76.5	19.3	80.3
ZB-5HT I	34.4	48.6	17.0	50.0	29.2	61.7	15.6	65.4	17.8	68.6	2.9	60.2	17.3	76.8	18.8	80.5
Rtx-5a	35.7	46.1	18.1	48.1	29.9	60.8	16.3	64.6	18.5	68.0	3.4	60.0	17.7	76.7	19.2	80.7
Rtx-5b	36.0	45.4	18.4	47.3	30.1	60.4	16.6	64.0	18.7	67.4	3.6	59.5	17.9	76.3	19.4	80.2
Rtx-5c	36.8	42.8	18.9	45.5	30.5	58.9	17.0	62.5	18.9	66.4	3.4	59.4	18.0	75.5	19.5	79.5
Rtx-5d	37.1	41.9	19.1	44.7	30.7	58.2	17.2	61.8	19.1	65.9	3.6	58.8	18.2	75.1	19.6	79.2
BPX-5	33.3	51.3	15.9	52.5	27.9	65.1	14.6	68.4	16.4	72.1	1.0	64.7	15.7	80.5	17.5	83.9

Table 2 Calculated excess enthalpies and entropies for each component on the 30 m × 0.25 mm × 0.25 µm

	2,6-dimethylphenol		2,6-dimethylaniline		1-decanol		n-tridecane		2-dodecanone		*N,N*-dicyclohexylamine		Methyl dodecanoate		n-hexadecane	
	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]	Enthaply [kJ mol^{-1}]	Entropy [J K^{-1}]
BPX-5	32.5	53.6	15.2	54.6	27.4	66.4	14.9	68.0	16.0	73.6	-0.2	67.0	15.4	81.7	17.3	84.9
ZB-5	35.0	47.4	17.7	48.6	29.5	61.6	16.3	64.5	18.2	68.4	4.1	58.3	17.4	77.2	19.1	80.7
Rtx-5	34.4	50.0	17.2	50.9	29.0	63.6	15.7	67.0	17.9	70.1	4.1	59.1	17.2	78.6	18.8	82.3

But when the linear velocity is increased, efficiency deteriorates to a greater extent for Rtx-5. BPX-5 has worse efficiency for every component even for alkanes. The H-u chart of tridecane is shown on Fig. 4.

The calculated excess enthalpies and entropies look similar, but on BPX-5 the values are a bit lower. These values are shown in Table 2. Only *N*,*N*-dicyclohexylamine is different, where these enthalpies are negative for BPX-5. This means the stationary phase is acidic and strong interactions are established with basic compounds of the sample. It is also confirmed by the lower enthalpy for 2,6-dimethylaniline. Entropies are the same, for every column and component. The enthalpies are much higher values than entropies. So enthalpy has the significant effect on the separation, not entropy.

These differences let us know which column should we use for the analysis. BPX-5 is acidic and could form H-bond with polar molecules. It has also low retention and efficiency. It should be used for simple matrix samples and for determining nonpolar compounds. ZB-5 is slightly acidic with a bit tailing, but it has the best resolution. Efficiency on low linear velocity is close to Rtx-5, but in high linear velocity it increases less. The excess enthalpies are the highest, so it has the weakest interaction with the molecules. This column is good for slightly basic components, but not recommended for strong bases. Rtx-5 has symmetric peaks for every component, even for *N*,*N*-dicyclohexylamine. Efficiency and resolution is also good. The excess enthalpy for *N*,*N*-dicyclohexylamine is similar to ZB-5, but the peak shape is not distorted. This means, we can use this column for many applications, with complex samples with different polarity compounds.

5 Conclusion

When we choose a column for our analysis, the manufacturers give us only the chromatogram, the plate number pro meter and retention indices for some compounds. Sometimes they determine the partition ratio or the asymmetry factor. But there are some parameters which should be shown.

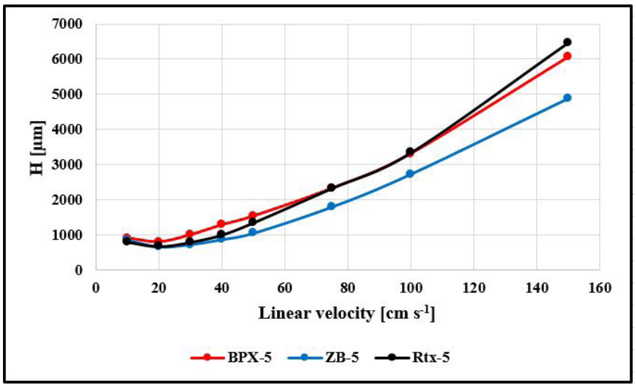

Fig. 4 The H-u chart of tridecane on 140 °C

Our test needs more measurements, but gives us more information about our column. The efficiency – linear velocity chart let us know on which column we can separate the compounds faster with less peak broadening. Also, we can choose the proper column, if we know something about the complexity of our sample. The excess enthalpies are very useful when choosing the right column. These values help us to find out the characteristic of the stationary phase and the strength of the interactions with measured compounds. For example, 5 % diphenyl – 95 % dimethyl polysiloxane is used many times for amine determination, but the acidic character of the stationary phase prevents it. So if we want to use the column for the proper task, we should also test these parameters.

Some of the tested columns were from different manufacturers, but recommended for the same task with similar stationary phase. Our results show us there are many differences, that is why we need more information to choose wisely the purchased column. Also the available similar column in a laboratory should tested to be used for the proper task.

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