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# Thermodynamic significance of boiling point correlations for alkylbenzenes in gas chromatography Extension of Trouton's rule<sup>☆</sup>

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## Abstract

A more exact thermodynamic interpretation of the empirical correlations between retention parameters of solutes (i.e. the relative retention times:  $\tau$  (s)) and their boiling points ( $T_B$ ) was established using Trouton–Hildebrandt–Everett's rule (the extension of Trouton's rule). These empirical correlations are known for C<sub>6</sub>–C<sub>12</sub> alkylbenzenes for low and medium polar stationary phases. A statistical analysis has been made to compare the description by the old and newly developed models. The exponential relation suggested earlier [*Chromatographia*, 44 (1997) 179–186] can be extended into a form consisting of two variables  $T_M$ ,  $T_B$ :

$$\tau_{(i)\text{corr.}} = AT_{M(i)} \exp(BT_{B(i)})$$

where:  $\tau_{(i)\text{corr.}} = (t_{R(i)} - t_0)/t_{R(st)}$ ,  $A = (t_0 \Phi / t_{R(st)}) \exp(-4.0)$ ,  $T_{M(i)} = T_{B(i)}^{(T_{B(i)}/T-1)}$ ,  $B = 4.0/T$ ,  $t_0$  is the column dead time,  $\Phi$  is the phase ratio,  $t_{R(st)}$  is the retention time of the standard compound,  $R$  is the gas constant,  $T$  is the column temperature, and subscript ' $i$ ' refers to the  $i$ th alkylbenzene. High correlation coefficients and small residual error indicate the superiority of the developed equation. Moreover, the residua show normal behavior, whereas curvature can be seen in the residua for earlier models. Validity of the approach proposed was confirmed through a comparison of the numerical values obtained from our computation (fitted values) with those stemming from theory. © 1999 Elsevier Science B.V. All rights reserved.

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## 1. Introduction

The development of quantitative structure–chromatographic retention relationships is one of the most important tools in the understanding of the mechanisms of retention, to predict retention data from physical and/or topological characteristics of

solute molecules and to calculate these characteristics from retention data [1,2]. The precision and generality are inversely proportional, i.e. one can achieve good description for a peculiar case (special compound class, homologues, etc.) or relatively bad description for general applications. Quantitative relationships can be classified into two groups, one having a definite physico-chemical basis and the other one lacking any physical rationale. The physico-chemical models are usually more general. They can be applied with more satisfactory results to a wide spectrum of practical cases. The purely empirical correlations can often be observed by chance. Even if the data are handled properly (with aid of statistical tests, using cross-validation, etc.) there is no guarantee of avoiding accidental correlations. Therefore, models having physico-chemical significance are superior to empirical ones. On the other hand, the empirical correlations usually prove to be more accurate within their validity range. Their practical application, however, is limited. A quantitative relationship with a firm physico-chemical basis and with an excellent predictive power is a much sought but rarely achieved idea.

The boiling point governs the retention on apolar and slightly to moderately polar stationary phases in gas-liquid chromatography. Hence, many empirical boiling point correlations were established for various compound classes and for stationary phases of different polarity [1–16].

Although there were some sporadic, limited trials to establish a physical basis to empirical correlations for several compound classes [1,2,8,13,17–19], to attribute physical meaning to equation constants in boiling point-correlations has not been attempted till now, apart from our earlier approach [20–22]. Recently, we attributed physical meaning to equation constants in relative retention data–boiling point correlations [20]. Similarly, we could calculate thermodynamic magnitudes from retention index–boiling point correlations [21,22] using some assumptions, e.g. the applicability of the Trouton's rule. The logical continuation of our efforts is to apply the more general and eventually more precise Trouton–Hildebrandt–Everett's rule.

Our aim was to develop further the thermodynamic model so that it can be characterized by both an acceptable physico-chemical basis, and a satisfac-

tory predictive power. These efforts are in conformity with a series of more extended thermodynamic papers [23–28].

### 1.1. Theory

Rearranging the basic equations of chromatography:

$$k_{(i)} = (t_{R(i)} - t_0)/t_0, \quad k_{(i)} = K_{(i)} \Phi \quad (1)$$

[where:  $k_{(i)}$  is the capacity factor, subscript '(i)' refers to the  $i$ th solute,  $t_{R(i)}$  is the retention time of the solute,  $t_0$  is the column dead time,  $K_{(i)}$  is the partition constant,  $\Phi$  is the phase ratio] the following equation was obtained:

$$\tau_{(i)\text{corr.}} = \tau_{(i)} - t_0/t_{R(\text{st})} = (t_0 \Phi) K_{(i)} / t_{R(\text{st})} \quad (2)$$

where  $\tau_{(i)} = t_{R(i)}/t_{R(\text{st})}$  is the relative retention time, and  $t_{R(\text{st})}$  is the retention time of the standard compound.

Using the basic equations of thermodynamics:

$$K_{(i)} = \exp(\Delta\mu_{p(i)}/RT) \quad (3)$$

$$\Delta\mu_p = \Delta H_p - T\Delta S_p \quad (4)$$

where  $\Delta\mu_p$  is the chemical potential of partitioning,  $\Delta H_p$  is the enthalpy of partitioning,  $\Delta S_p$  is the entropy of partitioning,  $R$  is the gas constant,  $T$  is the column temperature. Eq. (2) can be rearranged

$$\tau_{(i)\text{corr.}} = (t_0 \Phi) / t_{R(\text{st})} \exp(\Delta H_p / RT) \exp(-\Delta S_p / R) \quad (5)$$

The following assumptions have been made herewith: (i) transfer of solutes from the liquid stationary to the gaseous phase is a similar, although not identical process as vaporization of any given  $i$ th pure liquid, (ii) applicability of Trouton–Hildebrandt–Everett's rule, and (iii) the process 'solution' is regarded as a reverse process of vaporization, i.e. their thermodynamic quantities are equal to each other with an opposite sign.

The Trouton–Hildebrandt–Everett's rule can be given as [29]:

$$\Delta H_{\text{vap}(i)} = (4.0 + \ln T_{B(i)}) RT_{B(i)} \quad (6)$$

or in the form of:

$$\Delta S_{\text{vap}(i)} = (4.0 + \ln T_{\text{B}(i)})R \quad (7)$$

where  $\Delta H_{\text{vap}(i)}$  and  $\Delta S_{\text{vap}(i)}$  are the standard molar changes of enthalpy and entropy for the  $i$ th solute at the normal boiling point, respectively.

There is a considerable superiority of Trouton–Hildebrand–Everett’s rule over Trouton’s rule in physico-chemical terms. In fact, Trouton’s rule was based on a generalized observation as to the mutual relationship between the standard molar changes of enthalpy and entropy [ $\Delta H_{\text{vap}(i)}$  and  $\Delta S_{\text{vap}(i)}$ , respectively] at the normal boiling point ( $T_{\text{B}(i)}$ ) only for the vast majority of liquids. In this sense Trouton’s rule can even be considered as a very specific play of numbers. The Trouton–Hildebrand–Everett’s rule is, however, implemented with the important  $RT_{\text{B}(i)}$  term, which represents the molar volume of the vapor at the normal boiling point and, in this way, introduces a certain amount of physico-chemical relevance and, undoubtedly, also greater accuracy to the equation. It should be mentioned that the extension of Trouton’s rule can be considered as ‘one of the most remarkable generalizations in the whole of physical chemistry’ [29].

Introducing Eqs. (6) and (7) into Eq. (5) we obtain after rearrangement:

$$\tau_{(i)\text{corr.}} = (t_0 \Phi) / t_{R(\text{st})} \times \exp(-4.0) T_{\text{B}(i)}^{(T_{\text{B}(i)}/T)^{-1}} \exp(4.0/TT_{\text{B}(i)}) \quad (8)$$

or in a simpler form:

$$\tau_{(i)\text{corr.}} = AT_{\text{M}(i)} \exp(BT_{\text{B}(i)}) \quad (8a)$$

where  $A = (t_0 \Phi) / t_{R(\text{st})} \exp(-4.0)$ ,  $T_{\text{M}(i)} = T_{\text{B}(i)}^{(T_{\text{B}(i)}/T)^{-1}}$ , and  $B = 4.0/T$ . First a new variable, the modified boiling point ( $T_{\text{M}(i)}$ ) is calculated for each alkylbenzene. Then the minimum of squared differences of ( $\tau_{(i)\text{measured}}$  and  $\tau_{(i)\text{calculated}}$  by Eq. (8a)) is searched as a non-linear fit of two variables ( $T_{\text{M}(i)}$ ,  $T_{\text{B}(i)}$ ) and two parameters ( $A$  and  $B$ ) to be fitted.

## 2. Experimental

The experimental conditions (i.e. column characteristics and the working parameters) are described in detail elsewhere [9,10,20]. Relative retention times and boiling points were used, as given earlier [9,10].

Mesitylene (1,3,5-trimethyl benzene) has been chosen as standard according to the golden section of the serial numbers of alkylbenzenes in increasing boiling points. The experimental results concern exclusively for alkylbenzenes, but this is not mandatory. Equations similar to the derived one can be applied for other congeneric series, as well.

All calculations were made by the non-linear module of Statistica (Statsoft, OK, USA) program package. The error surface was complicated. The finding of local minima was avoided by beginning the non-linear fits by very different starting values of parameters to be fitted. It was expedient to use scaled variables for the exponential fits.

## 3. Results and discussion

Table 1 summarizes the statistical features of non-linear regressions together with the results of earlier models. In our preceding work we have developed physico-chemical basis for two empirical equations [20]:

$$\tau_{(i)\text{corr.}} = D \exp(ET_{\text{B}(i)}) \quad (9)$$

$$\tau_{(i)\text{corr.}} = F \exp(G/T_{\text{B}(i)}) \quad (10)$$

The physical meaning attributed to the  $D$ ,  $E$ ,  $F$ ,  $G$  constants is the following:

$$D = (t_0 \Phi / t_{R(\text{st})}) \exp(-85/R),$$

$$E = 85/RT,$$

$$F = (t_0 \Phi / t_{R(\text{st})}) \exp(\overline{\Delta H}_{\text{vap}}/RT),$$

and

$$G = -\overline{\Delta H}_{\text{vap}}/R.$$

It can be seen that the Eqs. (9) and (10) have little to do with the new equation. Still, Eq. (8a) is more similar to Eq. (9). However,  $B$  and  $E$  are not identical, and the difference between  $B$  and  $G$  is striking. Generally speaking, the new model provides better description than any of the earlier developed ones on each stationary phase investigated. The goodness of description can be seen in Fig. 1(a)–(d).

It can be seen in the figures that the goodness of

Table 1  
Regression summary for the refined (Eq. (8a)) and earlier models (Eqs (9 and 10))

	SIL <sup>g</sup>	DNP <sup>h</sup>	PEG <sup>i</sup>	BEN <sup>j</sup>
Fit by Eq. (8a)				
<i>N</i> (number of points)	18	14	14	18
<i>A</i> <sup>a</sup>	$6.479 \cdot 10^{-3}$	$0.1406 \cdot 10^{-3}$	$2.852 \cdot 10^{-3}$	$52.01 \cdot 10^{-3}$
Var [ <i>A</i> ] <sup>b</sup>	$4.293 \cdot 10^{-6}$	$0.04170 \cdot 10^{-6}$	$13.38 \cdot 10^{-6}$	$2650 \cdot 10^{-6}$
<i>B</i> <sup>a</sup>	$9.575 \cdot 10^{-3}$	$15.84 \cdot 10^{-3}$	$11.09 \cdot 10^{-3}$	$5.420 \cdot 10^{-3}$
Var [ <i>B</i> ] <sup>c</sup>	$0.5243 \cdot 10^{-6}$	$1.099 \cdot 10^{-6}$	$8.636 \cdot 10^{-6}$	$5.064 \cdot 10^{-6}$
Cov [ <i>A,B</i> ] <sup>d</sup>	$-1.500 \cdot 10^{-6}$	$-0.06767 \cdot 10^{-6}$	$-10.75 \cdot 10^{-6}$	$-115.8 \cdot 10^{-6}$
<i>R</i> <sup>e</sup>	0.99693	0.99766	0.97185	0.94899
<i>s</i> <sup>f</sup>	0.03233	0.02818	0.09050	0.07657
Fit by Eq. (9)				
<i>N</i> (number of points)	18	14	14	18
<i>A</i> <sup>a</sup>	$13.20 \cdot 10^{-6}$	$0.2437 \cdot 10^{-6}$	$5.730 \cdot 10^{-6}$	$120.3 \cdot 10^{-6}$
Var [ <i>A</i> ] <sup>b</sup>	$18.76 \cdot 10^{-12}$	$0.01319 \cdot 10^{-12}$	$56.06 \cdot 10^{-12}$	$14440 \cdot 10^{-12}$
<i>B</i> <sup>a</sup>	$25.71 \cdot 10^{-3}$	$34.73 \cdot 10^{-3}$	$27.68 \cdot 10^{-3}$	$19.77 \cdot 10^{-3}$
Var [ <i>B</i> ] <sup>c</sup>	$0.5515 \cdot 10^{-6}$	$1.156 \cdot 10^{-6}$	$8.957 \cdot 10^{-6}$	$5.157 \cdot 10^{-6}$
Cov [ <i>A,B</i> ] <sup>d</sup>	$-3.216 \cdot 10^{-9}$	$-0.1235 \cdot 10^{-9}$	$-22.40 \cdot 10^{-9}$	$-272.8 \cdot 10^{-9}$
<i>R</i> <sup>e</sup>	0.99681	0.99755	0.97113	0.94877
<i>s</i> <sup>f</sup>	0.03298	0.02880	0.09164	0.07673
Fit by Eq. 10				
<i>N</i> (number of points)	18	14	14	18
<i>A</i> <sup>a</sup>	$3.469 \cdot 10^4$	$2.782 \cdot 10^4$	$2.963 \cdot 10^4$	$0.2398 \cdot 10^4$
Var [ <i>A</i> ] <sup>b</sup>	$1.474 \cdot 10^8$	$1.086 \cdot 10^8$	$7.723 \cdot 10^8$	$0.05481 \cdot 10^8$
<i>B</i> <sup>a</sup>	$-4.562 \cdot 10^3$	$-4.499 \cdot 10^3$	$-4.488 \cdot 10^3$	$-3.566 \cdot 10^3$
Var [ <i>B</i> ] <sup>c</sup>	$2.380 \cdot 10^4$	$2.664 \cdot 10^4$	$16.65 \cdot 10^4$	$18.37 \cdot 10^4$
Cov [ <i>A,B</i> ] <sup>d</sup>	$-1.872 \cdot 10^6$	$-1.694 \cdot 10^6$	$-11.33 \cdot 10^6$	$-1.003 \cdot 10^6$
<i>R</i> <sup>e</sup>	0.99396	0.97017	0.95988	0.94550
<i>s</i> <sup>f</sup>	0.04534	0.09989	0.1077	0.07908

<sup>a</sup> Parameters to be fitted.

<sup>b</sup> First diagonal element of the covariance matrix, its square root is equal to the standard error of parameter *A*.

<sup>c</sup> Second diagonal element of the covariance matrix, its square root is equal to the standard error of parameter *B*.

<sup>d</sup> Off-diagonal element of the covariance matrix, its value is necessary for the correct error presentation [29].

<sup>e</sup> Correlation coefficient as defined by  $(1 - (\text{residual sum of squares}/\text{total sum of squares}))$ .

<sup>f</sup> Residual error (standard error of estimate).

<sup>g</sup> Silicon oil 550 stationary phase.

<sup>h</sup> Dinonylphthalate stationary phase.

<sup>i</sup> Polyethylene glycol 4000 stationary phase.

<sup>j</sup> Bentone 34 stationary phase.

description diminishes as the polarity of stationary phases increases. This remains in agreement with the role of specific interactions enhanced on polar phases. A particularly large scatter can be observed for the bentone 34 stationary phase. This scatter cannot be due to the experimental error only, but also due to the specific nature of this peculiar stationary phase. In other words, it is not only the boiling point, which governs solutes' retention on the GC polar stationary phases, but to a very consider-

able extent intermolecular interactions of the solute-stationary phase type also.

From the statistical evaluation of the results obtained it becomes evident, that except for Eq. (10), the best description of experimental retention is achieved for the data originating from the dinonylphthalate stationary phase.

Generally, it can be established that the refined model using the Trouton–Hildebrandt–Everett's rule describes the retention data slightly better than Eq.

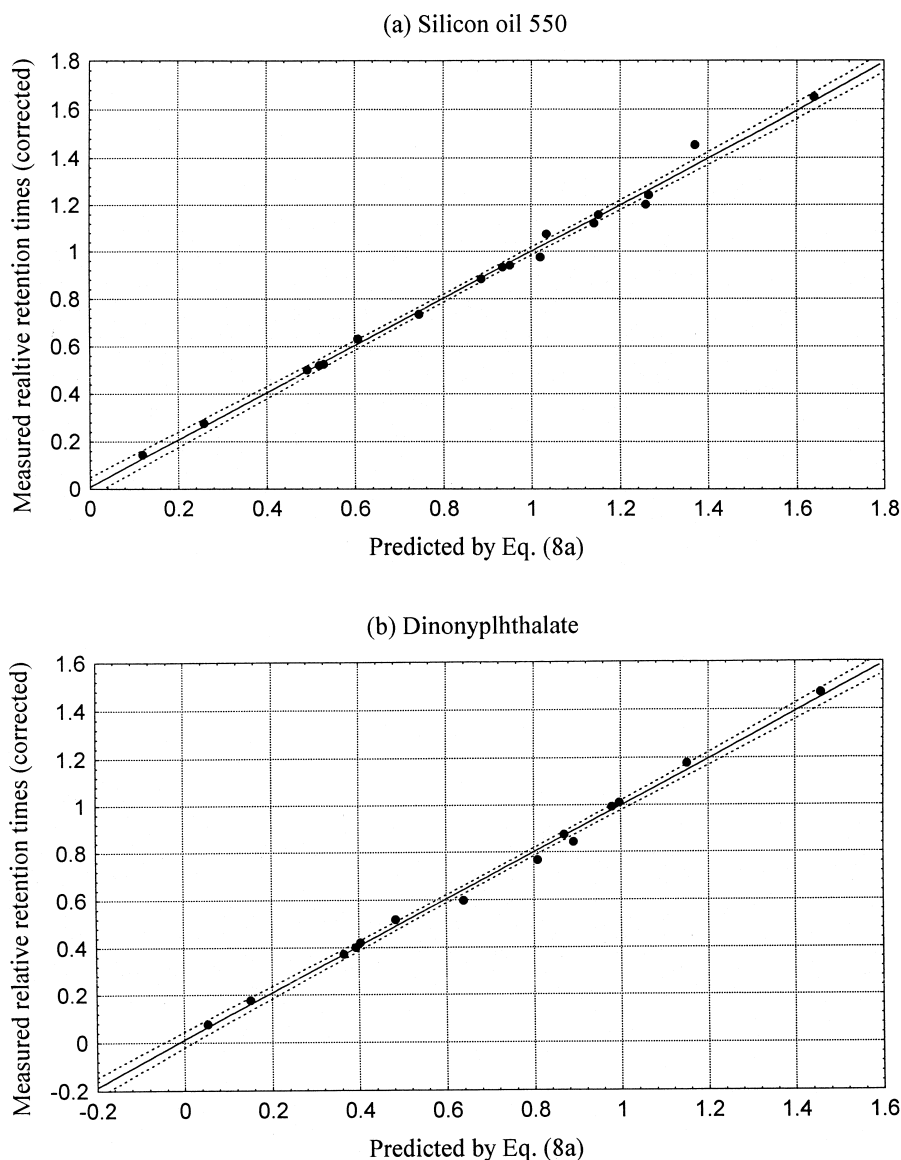


Fig. 1. Measured versus calculated (using Eq. (8a)) relative retention times (corrected) for stationary phases of different polarity. Dashed curves correspond to the 95% confidence intervals of the lines. (a) Silicon oil 550; (b) dinonylphthalate; (c) polyethylene glycol 4000; (d) bentone 34. (Continued on next page)

(9) and certainly much better than Eq. (10). This statement is based on the correlation coefficient ( $R$ ) and the standard error of the estimate ( $s$ ) unequivocally.

It should be noted that the  $T_M$  and  $T_B$  variables are intercorrelated, i.e.  $r(T_M, T_B) = 0.96892$  for silicon oil 550, 0.95945 for dinonylphthalate, 0.96729 for

polyethylene glycol 4000, and 0.97438 for bentone 34 ( $N = 14-18$ ). This intercorrelation seems to weaken the validity of the regression results. However, our regression equations in Table 1 clearly show a trend (as a function of the polarity of phases) which diminishes the chance of accidental correlation. The problem of correlated variables can be

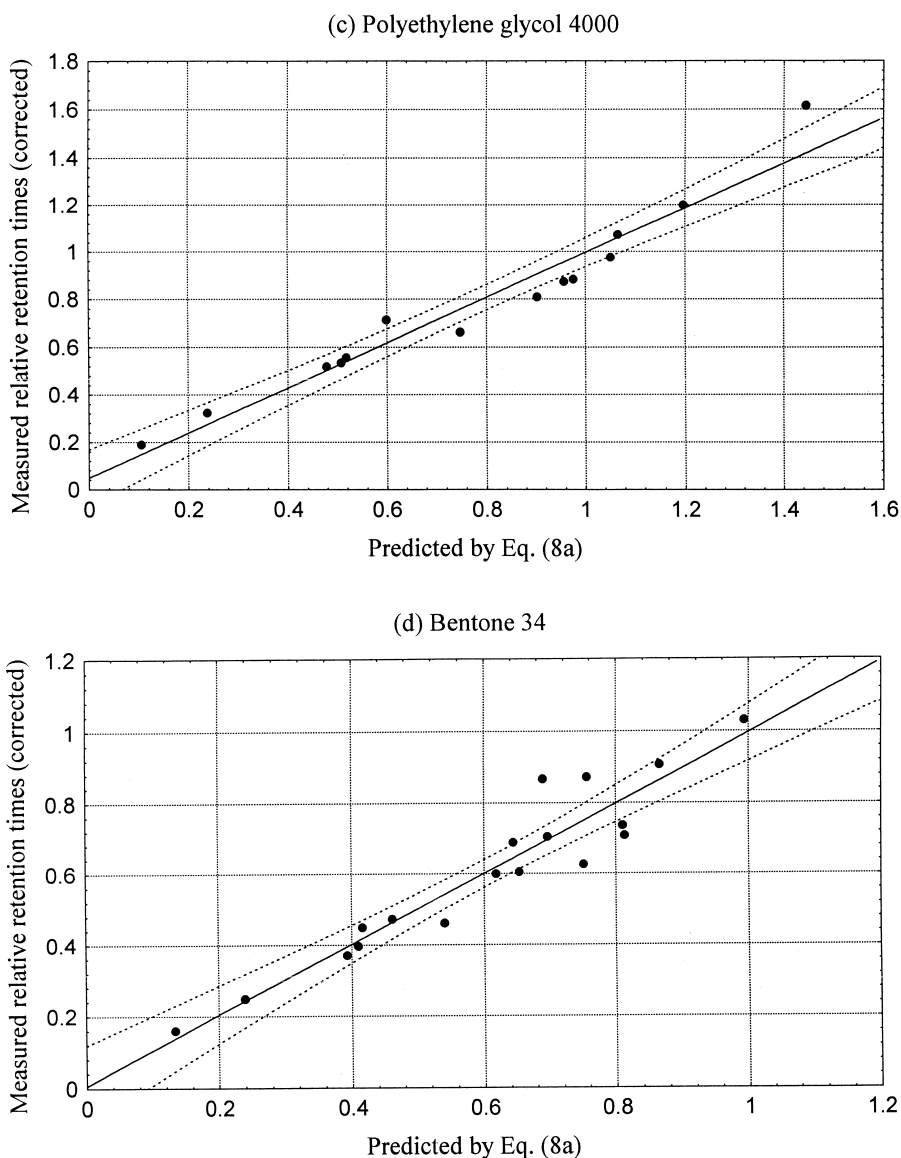


Fig. 1. (continued)

overcome by orthogonalization of variables. A principal component regression of  $T_M$  and  $T_B$  variables provides, as expected, a slightly worse description than a non-linear fit by the original variables. Some information loss arises during principal component regression and the model is linear in the latter case. The main disadvantage of this approach is that we cannot attribute physical meaning to the param-

eters determined by principal component regression.

Moreover, the predictive ability of our earlier equations becomes weaker as the polarity of stationary phases increased. The model developed by Trouton–Hildebrandt–Everett's rule is more general, it can be applied for more polar stationary phases as well. This can be seen in Table 2.

The theoretical parameter values (calculated from

Table 2  
Comparison of theoretical and fitted (using Eq. (8a)) parameters

	Silicon oil 550	Dinonylphthalate	PEG 4000	Bentone 34
<i>N</i> (number of points)	18	14	14	18
<i>A</i> fitted	$(6.479 \pm 2.07) 10^{-3}$	$(1.406 \pm 0.646) 10^{-4}$	$(2.852 \pm 3.66) 10^{-3}$	$(5.203 \pm 5.15) 10^{-2}$
<i>A</i> theoretical	0.01505	0.01813	0.05752	0.06040
<i>B</i> fitted	$(9.575 \pm 0.721) 10^{-3}$	$(1.585 \pm 0.105) 10^{-2}$	$(1.109 \pm 0.294) 10^{-2}$	$(5.420 \pm 2.75) 10^{-3}$
<i>B</i> theoretical	0.01832	0.01832	0.01832	0.01832

$t_0$ ,  $\Phi$ , Trouton–Hildebrandt–Everett’s constant,  $T$ , etc.) for the polar phases are close to the fitted ones within the error limits. The physico-chemical basis can be seen by a comparison of theoretical and fitted parameters using Eq. (8a). The agreement between the theoretical and fitted constants is acceptable in the case of  $B$  but unreliably large for the pre-exponential factor  $A$ . It is well known that the preexponential factor always has a larger error than the second parameter in the argument of an exponential. The deviation from theoretical values is caused by the so-called compensation effect [30]. It was shown earlier [30] with the help of computer simulations that the estimated parameters in an exponential fit are highly coupled, that is almost the same description can be achieved by somewhat smaller  $A$  and a larger  $B$  or vice versa. Another reason, which can cause a small deviation only, is the difference in column temperature and boiling temperatures. As the boiling points of alkylbenzenes are different, an averaged boiling point for all alkylbenzenes would have to be used as an isothermal working temperature in carefully planned future experiments. Strictly speaking, Trouton–Hildebrandt–Everett’s constant applies to spherical molecules only. Any deviation from spherical shape will necessarily cause a difference in the value of this particular constant.

In the course of derivating our new model we have never taken an advantage of the fact that the chromatographed compounds were alkylbenzenes. Therefore, it can be expected that the applicability of the relationship given by Eq. (8a) can also be extended to other, even polar groups of solutes analyzed by means of gas chromatography. Work is in progress to apply the new physicochemically based equation to other compound classes, like ketones and aldehydes.

#### 4. Conclusions

The relative retention time–boiling point correlation is not accidental but bears a firm physico-chemical importance. The application of Trouton–Hildebrandt–Everett’s rule makes it possible to develop a general model for apolar and polar stationary phases, as well. The newly developed model is not only more general but more precise also. Hence, it can be recommended for prediction purposes.

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