ELSEVIER

Journal of Chromatography A, 898 (2000) 103–109

JOURNAL OF CHROMATOGRAPHY A

www.elsevier.com/locate/chroma

Gas chromatographic study of the hydrogen bonding of aliphatic alcohols to tri-*n*-octylphosphine oxide

Reynaldo César Castells[a,b,*], Lilian Mónica Romero[a], Angel Miguel Nardillo[a,b]

[a]*División de Química Analítica, Facultad de Ciencias Exactas, Universidad Nacional de La Plata, 47 esq. 115, 1900 La Plata, Argentina*

[b]*CIDEPINT, 52 e/121 y 122, 1900 La Plata, Argentina*

Received 1 May 2000; received in revised form 27 July 2000; accepted 27 July 2000

Abstract

Retention volumes of 21 aliphatic alcohols were measured at five temperatures between 30 and 60°C in columns packed with different percentages of squalane or with solutions of tri-*n*-octylphosphine oxide (TOPO) in squalane coated on previously deactivated Chromosorb W. Experimental data fit to a 1:1 alcohol–TOPO association model, with association constants ranging from 26 to 59 $dm^3\ mol^{-1}$ at 45°C. Association constants follow the trend primary alcohols>secondary alcohols>tertiary alcohols, with minor differences between the members of each of these three groups. The association enthalpy for the 21 alcohols averages -21.8 kJ mol^{-1}, with a standard deviation of -1.3 kJ mol^{-1}

Keywords: Association constants; Thermodynamic parameters; Trioctylphosphine oxide; Alcohols

1. Introduction

Gas chromatography has been used profusely for the study of molecular associations. Interest in the subject was aroused after a presentation of Purnell during a chromatographic symposium held in 1966 [1], in which the author sketched several possibilities of the approach. A large number of papers was published in the following 15–20 years; reviews covering part of that period are available [2,3].

In the most common experimental approach, a solute X, which reacts rapidly and reversibly with an additive A to give a 1:1 complex, is chromatographed in columns containing different concentrations c_A of additive A in an inert solvent S (an alkane, if possible) as stationary phase. The apparent gas–stationary phase partition coefficient of X is given by:

$$K_L = K_L^*(1 + K_1 c_A) \quad (1)$$

where K_1 is the stoichiometric formation constant of complex AX in S and K_L^* is the partition coefficient of uncomplexed X. If K_L^* is assumed independent of c_A and therefore equal to the partition coefficient K_L^o of X between pure S and the gas phase, K_1 can be

*Corresponding author. División de Química Analítica, Facultad de Ciencias Exactas, Universidad Nacional de La Plata, 47 esq. 115, 1900 La Plata, Argentina. Fax: +54-221-427-1537.

E-mail address: rcastell@dalton.quimica.unlp.edu.ar (R.C. Castells).

0021-9673/00/$ – see front matter
PII: S0021-9673(00)00802-5

obtained from measurements of K_L at several values of c_A. An alternative proposed by Martire and co-workers [4–6] consists of measuring K_L for a solute that does not complex with A, assuming that the ratio of K_L in (A+S) to that in pure S is equal to the ratio K_L^*/K_L^o for the complexing solute; however, this equality cannot be confirmed experimentally and the effects of corrections of this type are unpredictable.

Most of the chromatographic complexing investigations were performed with sparingly or moderately polar solutes, such as H-bonding between haloalkanes and electron-donating additives or charge-transfer between aromatic molecules and additives bearing low electronic density groups. In studies involving more polar solutes (i.e., the type of solutes from which more diverse and intense interactions can be expected), complications related to difficulties in attaining infinite dilution conditions immediately became apparent; this was the type of problem met by Martire and co-workers [4–6] and by Cadogan and Purnell [7] during their investigations on H-bonding by aliphatic alcohols. Gas chromatography of these solutes in stationary phases of the type of inert solvent S suffers from two drawbacks. In the first place the strong solute adsorption onto siliceous surfaces (either regular or silanized) results in very marked peak asymmetry that persists even at the smallest sample sizes compatible with high sensitivity detectors. In the second place the strongly positive non-idealities of alkanol+alkane solutions lead to non-negligible solute adsorption at the gas–liquid interface; fortunately, peak asymmetry of this origin can be avoided by working with a sufficiently small sample size.

Retention times of asymmetric peaks are obviously sample size dependent; two methods have been used to obtain infinite dilution retention volumes from asymmetric peaks: the Martire and Riedl method [4–6] and Conder's method [8], modified by Cadogan and Purnell [7]. Both imply some type of extrapolation to zero sample size of retention times measured for samples of different size. Data reliability is obviously affected by corrections of this type; as a matter of fact, it was recently demonstrated that a minimum is shown by plots of alkanol retention times in squalane/Chromosorb W DMCS against peak area [9]. It was also shown in that paper that aliphatic alcohols (except for methanol and ethanol) elute as symmetrical peaks from columns packed with squalane coated on Chromosorb W deactivated by the method developed by Aue and co-workers [10,11]; peak maximum retention times are constant for samples ranging from a few microliters of vapour to fractions of 1 mm^3 of liquid. Comparison with results obtained statically in the absence of a solid support indicates that alkanols do not adsorb at the solid support–squalane interface, thus enabling us to express the retention volume at infinite dilution per gram of packing, V_N^o, by means of the equation:

$$V_N^o = K_L^o V_L + K_A^o A_L \quad (2)$$

where K_L^o and K_A^o represent the solute partition coefficient and its adsorption coefficient on the gas–stationary phase interface, in columns containing pure squalane as stationary phase. V_L and A_L are the stationary phase volume and the gas–liquid interface surface area, respectively, expressed per gram of packing.

An equation similar to Eq. (2) can be written to express V_N, the retention volume per gram of packing in a mixed A+S stationary phase:

$$V_N = K_L V_L + K_A A_L \quad (3)$$

where K_L and K_A retain their meaning as in Eq. (2), but now refer to the mixed stationary phase. Substituting K_L for its expression in Eq. (1):

$$V_N = K_L^* V_L + K_A A_L + K_L^* K_1 V_L c_A \quad (4)$$

Two assumptions have to be made in order to measure K_1 by means of Eq. (4): (a) $K_L^* = K_L^o$, and (b) $K_A = K_A^o$. If both assumptions can be justified experimentally, K_1 can be obtained from regressions of V_N against $V_L c_A$.

Trialkylphosphine oxides, R_3PO, are included in the stronger electron donors [12,13]. Gas chromatographically measured association constants of halomethanes with tri-*n*-octylphosphine oxide (TOPO) [14] are markedly larger than those of the same solutes with ethers, thioethers and tertiary amines [15–18]. Besides its electron-donating propensity, TOPO shows a very high dipole moment, about 4.5 D [19], and association with solutes with no possibility of H-bonding (tetrachloromethane, bromotrichloromethane), most probably resulting from classi-

cal electrostatic interactions, is by no means negligible. Application of the more sophisticated model of Martire [20], which considers the possibility of simultaneous interaction mechanisms, revealed that dipole interaction contributions to the association process are negligible in the case of molecules bearing active hydrogen atoms (haloforms, dichloromethane) [14].

TOPO–aliphatic alcohol associations are studied in the present work using squalane as the additive solvent and deactivated Chromosorb W as the solid support. Because of the very basic nature of TOPO, the elution of alcohols as symmetrical peaks from columns containing pure TOPO as stationary phase is highly probable, even using undeactivated solid supports. However, alcohol–TOPO interactions are so strong that elution would demand impracticably long retention times or very high temperatures: as a rule of thumb, alcohol retention times are doubled by adding only 1% (w/w) of TOPO to squalane.

2. Experimental

Deactivation of Chromosorb W 60–80 mesh was carried out by extracting with hot 6 *M* hydrochloric acid, washing to neutrality, drying at 120°C, coating with 4% (w/w) Carbowax 20M from chloroform, heating for 17 h at 270°C under nitrogen, and finally extracting for 30 h with methanol in a Soxhlet apparatus. Details can be found in the original publications of Aue and co-workers [10,11] and in our former paper [9]; we identify the final product as Chromosorb W CWX.

The melting point of TOPO as purchased (Eastman Kodak) was 51°C. A 10% solution of TOPO in *n*-hexane was shaken with 30% hydrogen peroxide in order to oxidize any phosphinous acids to the corresponding phosphinic acids [21] prior to separation; after three washings with water the solution was slurried with activated alumina, filtered and percolated through an alumina bed. The product crystallized from this solution melted at 53.5°C. Squalane (Hewlett-Packard) was used as received.

The density of pure squalane and of four solutions of TOPO in squalane (2.93_0, 5.99_4, 8.99_9 and 11.85_2%, w/w) was measured by pycnometry at 12–14 temperatures between 30 and 60°C. Experimental results were fitted to the equation:

$$\rho\ (\mathrm{g/cm^3}) = 0.81949 - 6.0899 \cdot 10^{-4} t + (4.16611 \cdot 10^{-4} + 4.239 \cdot 10^{-6} t) w_T \quad (5)$$

where t is temperature (°C) and w_T is TOPO % (w/w) in solution. The compositions of the packings used in chromatographic measurements are given in Table 1; alcohol retentions in pure squalane were formerly measured [9] in six different packings. Stationary phases were coated on Chromosorb W CWX from their solutions in *n*-hexane. Coated supports were packed into 1.5 m×0.53 cm I.D. stainless steel or 1.2 m×0.2 cm I.D. glass columns.

Chromatographic measurements were performed in a formerly described home-assembled apparatus [9] consisting of a Hewlett-Packard 5750 flame ionization detector and electrometer, a Hewlett-Packard 3396 integrator, and a water bath in which columns were immersed. Alcohol vapours, together with a small methane sample, were microsyringe injected; sample sizes were of the order of nmol, in the constant retention time region. Adjusted retention times were measured between the maxima of the solute (t_R) and methane (t_0) peaks; specific retention volumes were calculated using the equation:

$$V_g^o = jF_f(273.15/T_f)[(p_0 - p_w)/p_0](t_R - t_0)/w_L \quad (6)$$

where j is the James–Martin compressibility correction factor, F_f is the flow-rate measured at temperature T_f and pressure p_0 at the flowmeter, p_w is the water vapour pressure at T_f, and w_L represents the

Table 1
Packings employed in chromatographic measurements

% (w/w) stationary phase in the packings	TOPO molar fractions in the stationary phases	$c_A V_L$/mmol in the stationary phase at 45°C
4.03_2	0	0
4.01_3	0.0107	0.001013
3.99_3	0.0327	0.003089
4.03_3	0.0544	0.005196
5.96_0	0.	0
6.00_1	0.0109	0.001546
6.01_2	0.0327	0.004641
6.02_6	0.0571	0.081535

mass of the stationary phase contained in the column. Retention volumes were measured at five temperatures in the 30–50 or 40–60°C temperature intervals according to the solute vapour pressure.

3. Results and discussion

Small temperature differences (<0.2°C) when measuring retention volumes in different columns are unavoidable; data treatment by means of Eqs. (2) and (4) demands V_N values measured at exactly the same temperature. A correction for small differences was made by fitting the specific retention volumes measured in each column to the equation $\ln V_g^o = b/T + a$; the results obtained from these equations differed by less than 0.3% from the experimental values. Values of V_N were then calculated from:

$$V_N = V_g^o[w_L/(w_L + w_{SS})](T/273.15) \quad (7)$$

where w_L is the mass of the stationary phase coated on w_{SS} g of solid support. V_L (cm^3/g) for each packing was calculated from w_L and w_{SS} in combination with Eq. (5), and A_L by means of the relation A_L (cm^2/g) = 4500 − 14 700V_L, as reported previously [9].

Fundamental to the application of Eq. (4) is the previous demonstration that the partition coefficient of uncomplexed X in A+S mixtures is identical to the solute partition coefficient in pure S, and that the solute adsorption coefficient on the gas–liquid interface is not affected by the presence of A. It is expected, based on theoretical considerations, that none of these assumptions is strictly obeyed; for the purposes of the present work it will be sufficient to demonstrate the independence of K_L^o and K_A^o from c_A within the studied range of additive concentrations, according to the limitations imposed by our experimental errors.

Subtracting Eq. (2) from Eq. (4), the following expression is obtained:

$$V_N - V_N^o = (K_L^* - K_L^o)V_L + (K_A - K_A^o)A_L + K_L^* K_1 V_L c_A \quad (8)$$

Straight lines are obtained when $(V_N - V_N^o)$ values measured in columns containing pure squalane and three different TOPO concentrations in squalane as stationary phases, coated at both 4 and 6% (w/w) in the packings (a total of eight points), are plotted against $c_A V_L$. The results of the regression of data at 45°C are summarized in Table 2: intercepts (I) and slopes (P), with their respective standard deviations. Values of K_L^o and of infinite dilution activity coefficients at 45°C in squalane, γ_2^∞, partially taken from a former paper [22], were also included for later use.

Intercepts in Table 2 do not differ statistically from zero; according to Eq. (8) a zero intercept can be interpreted as indicating that both K_L^* and K_A are independent of the presence of TOPO. But it could also mean that linear relationships $K_L^* = K_L^o(1 + \alpha c_A)$, proposed by Liao et al. [23], and $K_A = K_A^o(1 + \beta c_A)$, where α and β are constants, were obeyed. However, slopes in this case would be given by:

$$P = K_L^o(\alpha + K_1 + 2\alpha K_1 c_A) + \beta K_A^o(A_L/V_L) \quad (9)$$

and although it is probable that plot curvature could not be detected, points corresponding to 4 and 6% (w/w) packings would fall on different lines since their (A_L/V_L) quotients differ by more than 60%. An additional possibility in relation to the interpretation of regression results is that they could be affected by the weight given to the two points at $c_A V_L = 0$; these two points were then removed, and regressions performed with the remaining six points also resulted in zero intercepts, with slopes differing by less than 2% from those of the first regression. Summarizing, it can be concluded from consideration of the above that K_L^* and K_A do not differ significantly from K_L^o and K_A^o, respectively, within the limits imposed by our experimental errors.

Association constants at 45°C, calculated from $K_1 = P/K_L^o$, are given in the last column of Table 2; their standard deviations were obtained from those of K_L^o and P by conventional error propagation calculations. Variation coefficients, $s(K_1)/K_1$, range from 0.026 to 0.048, with a mean of 0.04; this is an acceptable precision level for parameters calculated from data measured in several columns, similar to those of association constants measured by other experimental methods.

Alcohol association constants decrease in the order primary>secondary>tertiary, this being coincident with the trend found by Martire and co-workers [4,6]

Table 2
Intercepts (I) and slopes (P) resulting from fitting retention volumes to Eq. (8). Association constants (K_1, dm^3 mol^{-1}). Partition (K_L^o) and activity coefficients (γ_2^∞) in squalane. s, standard deviation. n.a., vapour pressure not available. $T = 45$°C

Solute		$I \pm s(I)$	$P \pm s(P)$	γ_2^∞	K_L^o	$K_1 \pm s(K_1)$
1	1-Propanol	−0.57±0.47	2.605±0.117	10.39	51.55±0.51	50.5±2.3
2	2-Propanol	0.05±0.19	1.132±0.046	9.06	29.49±0.19	38.4±1.6
3	1-Butanol	−0.88±1.27	7.420±0.318	9.61	151.7±1.3	48.9±2.1
4	2-Butanol	−0.75±0.60	3.349±0.150	6.44	92.14±0.50	36.4±1.6
5	2-Methyl-1-propanol	−1.15±0.95	5.847±0.238	8.76	107.1±0.9	54.6±2.3
6	2-Methyl-2-propanol	0.32±0.23	1.274±0.056	6.03	45.33±0.25	28.1±1.3
7	1-Pentanol	−2.67±2.39	24.96±0.60	8.56	429.9±3.5	58.1±1.5
8	2-Pentanol	−1.19±1.63	8.845±0.408	6.25	253.6±1.7	34.9±1.6
9	3-Pentanol	−1.27±1.47	9.378±0.368	4.51	267.8±1.8	35.0±1.4
10	2-Methyl-2-butanol	−0.61±0.68	4.046±0.169	4.09	156.8±1.1	25.8±1.1
11	1-Hexanol	−8.97±7.41	70.2±1.85	8.38	1187±9	59.3±1.6
12	3-Hexanol	−3.51±3.42	27.67±0.85	4.57	701.6±6.2	39.4±1.3
13	1-Heptanol	−9.73±21.4	189.7±5.4	8.55	3242±4	58.5±1.8
14	2-Heptanol	−9.44±10.9	79.29±2.72	5.79	1894±18	41.9±1.5
15	4-Heptanol	−8.95±10.4	71.83±2.60	4.53	1833±19	39.2±1.5
16	3-Octanol	−3.22±34.1	201.8±8.52	4.73	5138±39	39.3±1.7
17	4-Octanol	−3.93±27.6	190.9±6.90	4.46	4879±38	39.1±1.4
18	3-Methyl-3-heptanol	−0.22±16.6	88.61±4.14	3.07	3214±39	27.6±1.3
19	2,3-Dimethyl-2-hexanol	−0.86±14.7	78.12±3.67	n.a.	2608±22	30.0±1.4
20	3,4-Dimethyl-2-hexanol	−9.62±22.4	152.5±5.60	n.a.	3810±44	40.0±1.5
21	3,5-Dimethyl-3-hexanol	−7.40±11.6	63.54±2.90	n.a.	2196±25	28.9±1.4

and by Bhattacharyya et al. [24] in their studies on the association of isomeric propanols and butanols with tertiary amines, ethers and thioethers, with individual K_1 values markedly larger for associations with TOPO. The larger number of alcohols studied in the present work enables us to conclude that K_1 is hardly affected by chain length, branching or point of substitution within each of the three aforementioned classes of alcohols; this indicates an important differences in relation to the solution process, since activity coefficients in squalane are strongly affected by the characteristics of the alcohol carbon chain. These trends are shown in Fig. 1, where the free energies of complexation, $\Delta G_1 = -RT \ln K_1$, are plotted against partial molar excess free energies of solution in squalane, $G_2^{E,\infty} = RT \ln \gamma_2^\infty$; the idea behind this figure, that some kind of correlation could exist between K_1 and γ_2^∞, must be discarded.

Literature reference to alkanol–TOPO complexing is scarce. Huyskens et al. [25] report $K_1 = 190$ dm^3 mol^{-1} from calorimetric measurements for methanol–TOPO in cyclohexane at 25°C. The difference from our results can be justified in terms of the temperature difference and because methanol is the

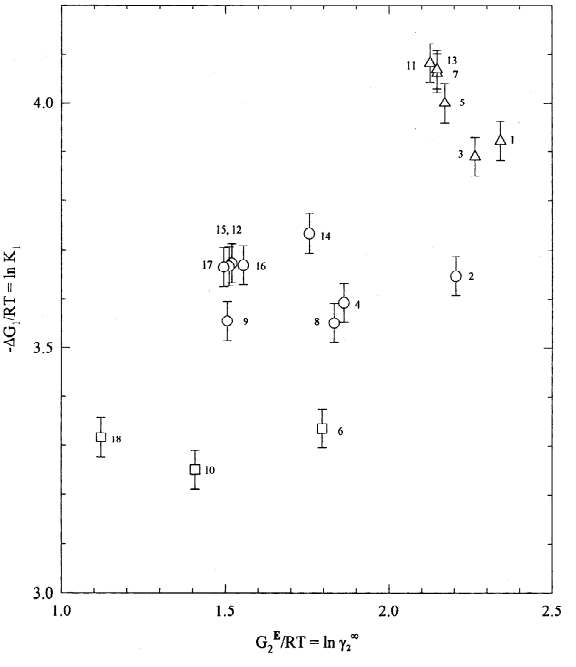

Fig. 1. Free energy of complexation (ΔG_1) versus partial molar excess free energy of solution in squalane (G_2^E) at 45°C. Triangles, primary alcohols; circles, secondary alcohols; squares, tertiary alcohols. Solute numbers: see Table 2.

strongest acid in the alkanol series, with $K_a = 8 \cdot 10^{-16}$, versus $8 \cdot 10^{-17}$ for 1-propanol and 1-butanol and 10^{-19} for 2-propanol [26]. Abraham et al. [13,27,28] compiled association constants for a large number of acids and bases in tetrachloromethane at 25°C and developed general acidity and basicity scales; complexing constants for a given acid–base pair can in principle be calculated from the parameters given in their tables. Although TOPO is not included in the compilation, parameters are given for triethylphosphine oxide and for six of the alkanols of our list; calculated association constants are 45 dm^3 mol^{-1} for methanol and from 16 to 24 dm^3 mol^{-1} for the rest of the alcohols. However, tetrachloromethane interacts non-negligibly with trialkylphosphine oxides, and $K_1 = 0.30$ dm^3 mol^{-1} was measured against TOPO in squalane at 60°C [14] (compared with 8.92 dm^3 mol^{-1} for chloroform under the same conditions); this impairs a fair comparison between calculations using Abraham's parameters and our experimental results.

Complexation enthalpies, calculated from $\ln K_1$ vs. T^{-1} plots, show little variation between solutes, averaging -21.8 kJ mol^{-1} with a standard deviation of 1.3 kJ mol^{-1}; since uncertainties in ΔH_1 are about 5–10% (as calculated by the method developed in Ref. [29]), trends in complexation enthalpies cannot be detected. Huyskens et al. [25] measured -27.3 kJ mol^{-1} for the pair methanol–TOPO, a result larger than ours but not unexpected on account of the stronger acidic character of methanol.

4. Conclusions

By using a specifically deactivated support, Chromosorb CWX as developed by Aue et al., for alcohols analysis, alkanols chromatographed in squalane or squalane + TOPO stationary phases do not adsorb at the solid support–liquid phase interface; infinite dilution is attained and correction of retention times by means of cumbersome and unreliable methods becomes unnecessary, thus diminishing errors and the demands of experimental work. Both the partition coefficient of uncomplexed alcohol (K_L^*) and its adsorption coefficient at the gas–liquid interface (K_A) in the presence of TOPO are apparently (i.e., within the possibilities of experimental detection) indistinguishable from their respective counterparts in pure squalane (K_L^o and K_A^o, respectively). However, changes in K_L^* and K_A with additive concentration could be masked by the strong effects that the addition of small amounts of TOPO has on retention; less basic additives are being investigated. The availability of deactivation procedures analogous to that of Aue et al. but specific for other types of polar solutes (amines, for instance) would permit the study of chemical equilibria out of reach of the chromatographic method.

Acknowledgements

This work was sponsored by CONICET (Consejo Nacional de Investigaciones Científicas y Técnicas de la República Argentina), by Agencia Nacional de Promoción Científica y Tecnológica (contract BID 802/OC-AR) and by CICPBA (Comisión de Investigaciones Científicas de la Provincia de Buenos Aires).

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