

Effect of Temperature on Specific Retention Volumes of Selected Volatile Organic Compounds Using the Gas - Liquid Chromatographic Technique Revisited

Edison Muzenda and Ayo S. Afolabi

Abstract—This paper is a continuation of our interest in the influence of temperature on specific retention volumes and the resulting infinite dilution activity coefficients. This has a direct effect in the design of absorption and stripping columns for the abatement of volatile organic compounds. The interaction of 13 volatile organic compounds (VOCs) with polydimethylsiloxane (PDMS) at varying temperatures was studied by gas liquid chromatography (GLC). Infinite dilution activity coefficients and specific retention volumes obtained in this study were found to be in agreement with those obtained from static headspace and group contribution methods by the authors as well as literature values for similar systems. Temperature variation also allows for transport calculations for different seasons. The results of this work confirm that PDMS is well suited for the scrubbing of VOCs from waste gas streams. Plots of specific retention volumes against temperature gave linear van't Hoff plots.

Keywords— Specific retention volume, Waste gas streams, specific retention, infinite dilution, abatement, transport.

I. INTRODUCTION

THIS paper is our latest attempt to explain the temperature dependence of the solubility of volatile organic compounds in terms of specific retention volumes and the resulting infinite dilution activity coefficients. The initial and some aspects of this work were presented [1] and subsequently accepted for publication [2]. These were brief and short page restricted papers. In this work an expanded detailed explanation is given. Polymeric solvents may play an important role in key chemical operations such as separation processes. Most of the known organic solvents such as polydimethylsiloxane can be used to solve air pollution problems due to their relatively high volatilities. In order to use polymeric solvents such as PDMS to effectively scrub volatile organic compounds from contaminated air streams, it is essential to know how they interact with different solutes.

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The important measure of this property is given by the specific retention volume (V_g^o) or the activity coefficient at infinite dilution (γ_i^∞). Specific retention volumes can be used through the reduction of infinite dilution activity coefficients to design separation processes where the trace components or impurities have to be removed. The GLC can be used to measure specific retention volumes of volatile organic compounds in PDMS because of the small vapour pressure of the later. The procedure of the measurement in particular, the effect of flow rate of carrier gas, effect of sample size, and liquid loading were previously investigated [1] - [4].

The specific retention volume is defined as the net retention volume per gram of stationary phase at 0°C. This is very important because it allows the comparison of retention data obtained at different temperatures with different weight of stationary phase. Specific retention volumes were introduced by [5]. He suggested their use in place of partition coefficients in vapour identification. Ref [5] measured specific retention volumes for a series of alcohols, aromatic hydrocarbons, and esters in silicone 702 – fluid and tritoyl phosphate. Ref [6] measured specific retention volumes using GLC for polydimethylsiloxane – hydrocarbon systems at 298, 313 and 328K. The carrier gas flow rate was varied from 18 to 120 cm³ per minute and it was found to have no effect on the specific retention volumes. Ref [7] obtained specific retention volumes for polydimethylsiloxane – hydrocarbon systems in four columns with different liquid loading. Experimental temperature and flow rate were varied from 298 – 343K and 70 to 120ml/min respectively. Ref [8] reported replicate gas – liquid chromatographic based specific retention volumes, activity coefficients and interaction parameters of ten solutes with polydimethylsiloxane at 303K.

Though inconclusive, the effects of temperature on retention data in particular Kovats indices, have been investigated and debated for a long time [9]. Almost linear dependence of retention data for non polar solutes on non polar phases have been reported [9]. The Antoine type (which is non linear) shows better performance for wide temperature range for systems involving non polar solutes on polar

stationary phases [10]. There have also been numerous studies of temperature effects on solute retention in reversed phase liquid chromatography (RPLC). Linear Van't Hoff plots were observed in the typical RPLC systems as reported and cited [11]. In the studies reported in [11], enthalpies of solute transfer from the mobile phase to the stationary phase were calculated from the van't Hoff plots. Non linear plots are often observed when the temperature range is more than 318K.

Usually the temperature dependency of the specific retention volume is expressed as

$$\ln V_s^0 = P \frac{1}{T} + Q \quad (1)$$

Where

$$P = - \frac{\Delta H^s}{R} \quad (2)$$

$$Q = \ln \frac{273 R}{M_s} + \frac{\Delta S^s}{R} \quad (3)$$

Where R is the universal gas constant, M_s is the molecular mass of the stationary phase. ΔH^s and ΔS^s are respectively, the standard molar enthalpy and entropy of solution for the transfer of a mole of solute, from the ideal gas where its partial pressure is 1 atm, in the stationary phase, where its mole fraction is $x = 1$.

II. METHODOLOGY

The apparatus used have been described previously [3], [4], [11]-[13]. The carrier gas was helium. A constant sample size of 0.1 μ l was injected into the columns at a constant flow rate of 35.97ml/min. Measurements were varied from 303K to 423K. Special care was exercised to control and determine the system temperature accurately.

A. Column Preparation

Polydimethylsiloxane (PDMS) was coated into Chromosorb P, AW - DMCS or Chromosorb W, AW - DMCS (acid washed, dimethylchlorosiloxane treated) from a solution in chloroform. Details of column preparation are shown in Table 1.

TABLE I
DESCRIPTION OF COLUMNS

Properties	Column 1	Column 2	Column 3	Column 4
Length, m	1	1	1	1
Chromosorb W or P,g	6.158	7.291	7.186	4.86
PDMS,g	0.688	0.825	0.804	0.54
Wt % PDMS	10.05	10.16	10.06	10
Viscosity of PDMS, cp	5	10	50	500
Mw of PDMS	760	1000	3200	13000

B. Materials

The materials used have been described previously [3], [4], [11]-[13]. The thirteen solutes used in this work were coded numerically as in Table 2 for simplicity.

TABLE II
SOLUTE DESCRIPTION AND CODE

Code no.	Compound	Code no.	Compound
1	chloroform	8	xylene
2	n-pentane	9	diethylether
3	n-hexane	10	butylacetate
4	n-heptane	11	Isobutyl methyl ketone
5	acetone	12	triethylamine
6	toluene	13	ethyl lmethyl ketone
7	cyclohexane		

C. Calculations

The primary GLC data are the retention time, that is the time taken by the component to traverse the column and the retention volume V_R , the volume of carrier gas passing through that time. The net retention volume is defined as in equation (4), where V_{gas} is the volume of gas in the column (gas hold-up). The gas hold up volume is a measure of total volume of space available to the mobile phase in the system, that is the column dead volume, injector and detector volumes and volumes of any connecting tubing for example from column to detector. In a well designed system, the extra dead volume is small compared to the column void volume.

$$V_N = V_R - V_{gas} \quad (4)$$

In gas-liquid columns the gas hold-up contributes 1 to 10 percent of the total retention volume. The homologous series method was used in this work because of the sensitivity of the flame ionization detector at low concentrations of the organic compounds. The time the non sorbed molecule take to traverse through the column is given by equation (5)

$$t_M = \frac{t_{R_1} t_{R_3} - t_{R_2}^2}{t_{R_1} + t_{R_3} - 2t_{R_2}} \quad (5)$$

The retention time t_R is made up of t_M , the time the solute spends in the mobile phase and t_s , the time the solute spends in the stationary phase. Separations are due to different times solutes spend in the stationary phase. The adjusted retention volume is given by equation (6).

$$V_R' = F_o t_R' = V_R - V_M \quad (6)$$

The corrected retention volume, V_R^o which takes into account the compressibility of the gaseous mobile phase is given by equation (7).

$$V_R^o = j_3^2 V_R \quad (7)$$

The factor j_3^2 in equation (7) corrects for the influence of pressure along the column and is given by equation (8).

$$j_3^2 = \frac{3}{2} \frac{\left[\left(\frac{P_i}{P_o} \right)^2 - 1 \right]}{\left[\left(\frac{P_i}{P_o} \right)^3 - 1 \right]} \quad (8)$$

The relationship between the net retention volume V_N and the carrier gas flow rate at the column outlet F_o and the retention times t_R and t_M for absorbed solute and carrier gas respectively is given by equation (9).

$$V_N = j_3^2 F_o (t_R - t_M) \quad (9)$$

The specific retention volume, corrected to 0°, is given by equation (10).

$$V_g^o = \frac{273.15 V_N}{W_s T_c} \quad (10)$$

In equations (7-10), j_3^2 is the gas compressibility factor, F_o is volumetric carrier gas flow rate at column outlet temperature and pressure, ml/min; $t_R - t_M$ is the retention time, i.e., the time difference between air and solute peaks, min; T_c is column temperature, °K; W_s is weight of polymer in the column, g; p_i is inlet and p_o is outlet pressure. The use of the flame ionization detector permitted the application of the mathematical air peak method to approximate air peak maximum. To account for the gas holdup in the column, the retention time was taken as the difference between the maxima of the air and solute peaks [14].

III. RESULTS AND DISCUSSION

A. Variation of specific retention volumes from literature findings

TABLE III
VARIATION OF SPECIFIC RETENTION VOLUMES FROM LITERATURE FINDINGS (PDMS MW \approx 3200)

Code	Temp (K)	Specific retention volumes		%	Source
		This work	Literature		
1	303	188.2	181.7	3.4	[8]
	313	47.8	47.7	0.3	[8]
	333	24.9	24.8	0.7	[8]
3	303	192.0	179.2	7.1	[8]
	313	126.2	124.2	1.6	[16]
	333	58.8	60.9	3.5	[5]
4	303	508.3	482.5	5.3	[6]
	313	294.6	290.8	1.3	[15]
	333	146.8	144.3	1.7	[13]
6	303	796.5	791.1	0.7	[8]
	313	529.1	516.6	2.4	[6]
	323	300.2	269.2	11.5	[6]
7	303	336.8	315.1	6.9	[6]
	333	103.4	106.0	2.5	[5]
8	303	2645.6	2654.6	0.3	[15]
	313	1223.6	1187.5	3.0	[6]
	333	516.3	536.5	3.8	[16]

The solute specific retention volumes reported in Table 3 were calculated from corrected peak retention times using the well known expression of [5]. The retention times used in the calculation of specific retention volumes were averages of five measurements. Individual values of retention times were found to vary by no more than 1% in all cases. Specific retention volumes reported in this work compare very well with literature findings. The successful comparison gives an indication of the GLC as a rapid, simple and accurate method for studying the thermodynamics of the interaction of a volatile solute with a non volatile solvent.

B. Temperature dependence of specific retention volumes

TABLE IV
VOC SPECIFIC RETENTION VOLUMES IN PDMS (Mw \approx 760)

VOC	Temperature (K)							
	303	313	323	333	353	373	393	423
1	159.0	105.6	72.4	49.5	25.8	13.3	9.2	4.5
2	61.1	45.1	34.2	24.94	15.3	9.9	7.0	4.3
3	173.5	124.7	82.7	58.8	32.7	18.7	11.9	6.5
4	461.7	294.6	191.8	127.2	63.6	36.3	21.6	10.8
5	59.9	44.4	28.9	18.9	14.1	9.9	7.8	4.3
6	782.0	471.2	300.2	199.2	95.4	50.2	28.8	14.5
7	338.0	219.5	148.1	100.9	55.8	33.2	18.9	10.5
8	2443.2	1268.2	858.6	512.1	187.4	93.4	60.1	23.3
9	61.5	42.5	30.5	23.2	13.2	9.9	6.7	3.3
10	1293.2	717.7	411.5	243.7	104.1	50.8	25.1	15.0
11	627.4	318.1	188.8	120.5	64.2	33.2	17.5	8.3
12	414.0	264.6	168.8	111.2	53.1	29.0	15.2	7.3
13	150.8	100.5	66.1	46.2	33.9	22.7	17.0	8.8

TABLE V
VOC SPECIFIC RETENTION VOLUMES IN PDMS (Mw \approx 1000)

VOC	Temperature (K)							
	303	313	323	333	353	373	393	423
1	188.2	132.5	88.7	64.2	33.5	17.8	12.5	6.9
2	75.2	55.9	39.7	29.1	15.9	9.7	6.1	3.9
3	202.6	142.1	95.9	67.0	32.6	19.1	11.6	6.4
4	545.8	361.4	231.8	154.0	74.7	37.4	21.7	11.2
5	73.3	50.1	35.3	26.2	13.2	7.7	4.5	2.2
6	796.5	497.0	350.0	193.2	92.9	49.9	28.1	14.1
7	355.2	234.2	154.7	110.0	56.2	31.4	17.8	9.7
8	2688.5	1454.8	671.5	426.9	179.0	94.1	48.2	21.8
9	76.7	58.0	40.8	30.9	18.7	12.0	8.2	4.4
10	1584.5	852.1	552.0	346.7	141.8	67.2	34.1	20.2
11	763.1	473.4	298.5	185.7	86.7	42.4	23.6	11.3
12	446.3	291.1	193.4	128.7	62.6	33.7	18.2	7.6
13	170.9	121.7	82.6	62.7	41.0	31.3	23.0	12.5

TABLE VI
VOC SPECIFIC RETENTION VOLUMES IN PDMS ($M_w \approx 3200$)

VOC	Temperature (K)							
	303	313	323	333	353	373	393	423
1	198.2	131.4	89.6	61.6	34.5	18.2	11.7	6.5
2	78.1	56.4	40.7	29.2	15.8	10.8	7.7	4.4
3	205.7	140.1	95.4	65.4	34.8	18.4	12.7	6.5
4	547.0	348.3	222.1	146.8	69.2	35.5	22.2	12.3
5	65.1	42.3	28.3	20.9	12.0	8.4	5.2	2.8
6	813.2	500.0	304.4	175.3	95.4	51.3	36.5	14.2
7	349.1	158.3	158.3	110.2	57.8	32.6	18.7	10.2
8	2645.5	1129.9	795.4	516.3	213.8	111.8	57.7	26.2
9	75.8	50.5	36.8	27.1	16.1	10.3	6.7	3.7
10	1600.2	923.6	553.2	335.6	137.6	65.2	33.5	19.7
11	793.8	485.0	301.1	188.3	85.9	42.6	23.7	11.4
12	470.9	297.8	191.7	130.0	64.2	34.7	19.5	8.1
13	180.1	129.8	86.6	65.1	34.2	25.5	18.5	10.0

TABLE VII
VOC SPECIFIC RETENTION VOLUMES IN PDMS ($M_w \approx 13000$)

VOC	Temperature (K)							
	303	313	323	333	353	373	393	423
1	191.0	126.2	85.9	59.1	33.4	17.6	12.4	6.8
2	69.9	47.8	35.4	26.8	16.7	10.4	7.3	4.4
3	192.0	136.2	88.0	57.1	30.5	18.5	10.7	6.8
4	508.3	301.3	202.0	134.3	66.2	35.6	21.0	10.7
5	68.8	48.3	35.4	26.3	12.4	7.2	4.3	2.0
6	852.3	529.1	370.6	201.4	100.0	52.3	30.4	15.1
7	336.8	230.4	144.2	103.4	56.7	32.0	18.4	9.9
8	2452.5	1223.6	688.6	403.4	203.4	95.6	49.6	22.3
9	79.8	55.3	39.0	27.8	16.7	10.4	7.3	4.0
10	1704.0	912.6	588.7	367.6	150.1	70.3	37.2	21.1
11	814.6	503.3	314.6	193.4	88.6	44.2	24.4	11.9
12	469.1	306.5	201.3	131.7	64.8	36.5	18.8	8.0
13	173.6	124.8	88.7	59.9	38.6	28.4	19.3	10.7

The specific retention volumes of chloroform, pentane, hexane, heptane, acetone, toluene, cyclohexane, xylene, diethyl ether, butyl acetate, isobutyl methyl ketone, triethylamine and ethyl methyl ketone were measured by injecting a constant amount of sample of 0.1 μ l into the four columns. Chromatographic retention data from variable temperature runs may be used to estimate thermodynamic properties according to the well known van't Hoff relation [10] using (11).

$$\ln k' = -\Delta H / RT + \Delta S / R + \ln \phi \quad (11)$$

Where ϕ is the phase volume ratio and R is gas constant.

The linear portions of these plots give enthalpies ($\Delta H / RT$) and entropies (ΔS) for the transfer of one mole of solute into the stationary phase. As reported [1], [2] the van't Hoff plots from data in Tables 4 to 7 are linear, and the regression correlation coefficients are greater than 0.999 in all cases. Specific retention volumes decreased with decrease in temperature. The trends obtained here are similar to those observed by [10], [17]-[23].

The choice of the optimum temperature for the absorption and stripping processes requires knowledge of temperature dependence of the activity coefficient. Infinite dilution activity coefficients calculated from these specific retention volumes show that the higher the temperature the higher the activity coefficients [24]. This tendency is very favourable for desorption, because higher values at higher temperature would ease regeneration.

IV. CONCLUSION

Specific retention volumes for 13 volatile organic compounds in polydimethylsiloxane were measured at varying temperatures. This allows estimation of the specific retention volumes and hence infinite dilution activity coefficients at temperatures not considered in this study. The measurements were highly reproducible with relative standard deviation and coefficient of variation in the determination of specific retention volumes of 0.00013 and 0.013 respectively. This can be applied to measure thermodynamic interactions of volatile organic compounds with polymeric solvents such as biodiesel, squalane, biphenyl, glycol polymers and higher alkanes. These are some of the solvents of interest to us in the scrubbing of volatile organic compounds.

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