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Measurement of Activity Coefficients at Infinite Dilution of Chlorinated Solvents in Commercial Polyethylene Glycol Ethers

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Among the suggested scrubbing liquids, alkylene glycol dialkyl ethers are especially suitable because of their favorable properties. Activity coefficients at infinite dilution are reported in the literature only for single absorption liquids, but generally in industrial applications stabilized mixtures of these substances are used. In this work two commercial products were considered: Genosorb 300 and Genosorb 1843, mixtures of polyethylene glycol dimethyl ethers and of polyethylene glycol dibutyl ethers, respectively. Gas–liquid chromatography was chosen for the experimental determination of the activity coefficients at infinite dilution, in the temperature range between 42 and 90 °C, of commonly used organic solvents, belonging in particular to the halocarbon class.

Introduction

The contribution of solvents and other volatile compounds to environmental pollution is becoming more and more significant. Awareness of this fact has brought about the growth of a body of laws for limiting the emissions.

Various techniques for prevention, abatement, and recycling of the outcoming vapors are therefore becoming available (Mueller and Ulrich, 1991; Schiffbauer, 1990). Since combustion often involves additional fixed costs (postcombustor) and condensation requires severe operating conditions to meet the emissions limits, absorption seems to be the most advantageous strategy. The increased interest in the absorption process is due both to the identification of new absorption liquids characterized by high chemical and thermal stability, low viscosity, and low vapor pressure (Geisthardt et al., 1989) and to the use of absorption columns equipped with packings that make it possible to achieve high efficiencies even with very low liquid loads (Meier et al., 1977).

The design of absorption equipment requires equilibrium data (activity coefficients at infinite dilution and Henry coefficients) that are reported in the literature only for pure absorption liquids (Schiller and Gmehling, 1992). The washing liquids are pure liquids only in a few applications; in industrial applications commercial mixtures are generally used.

In this work two commercial high-boiling products are considered (Genosorb 300 and Genosorb 1843, Hoechst). They are mixtures of polyethylene glycol dimethyl ethers and polyethylene glycol dibutyl ethers, respectively. Gas–liquid chromatography was chosen for the experimental determination of the activity coefficients at infinite dilution of commonly used organic solvents, in particular of the chlorinated class.

Analysis Method and Fundamental Relations

For the selection of the most suitable absorbing liquids, with regard to the solvent to be recovered, and the design of the purification apparatus, equilibrium data are required. Among the various techniques available (Tiegs et al., 1986; Dallinga et al., 1993) for the evaluation of the activity coefficients at infinite dilution, the gas chromatographic elution method, described in detail by Littlewood (1970) and applied by other authors (Schiller and Gmehling, 1992; Eckert et al., 1981; Weidlich and Gmehling, 1987), was chosen in this work. In comparison to the dilutor technique or the static methods, gas–liquid chromatography (GLC) offers various advantages, among them measurement of the equilibrium data at infinite dilution over a wide range of temperatures and in a rather short time (Tiegs et al., 1986).

Determination of the activity coefficients at infinite dilution requires measurement and control of the following parameters: net retention time of eluted solute, operating temperatures, inlet and outlet chromatographic column pressures, carrier gas volumetric flow, and mass of the stationary liquid on the inert solid support (carrier material). The net retention time of eluted solute is given by the difference between the retention time of the solute (elapsed time between the injection of the sample and the read of the peak maximum) (solute peak) and the "dead" time required for an inert gas, such as air, to pass through the chromatographic column.

The activity coefficient at infinite dilution can be determined from the quantity actually measured, the so-called "specific retention volume V_g^0, corrected to 0 °C". This is related to the experimentally determined parameters by the equation (Conder and Young, 1978)

$$V_g^0 = \frac{(t_R - t_A) \times 273.15F_A}{m_L}\left(\frac{P_{fm} - P_w}{P_oT_{fm}}\right)\left[\frac{3}{2}\frac{(P_i/P_o)^2 - 1}{(P_i/P_o)^3 - 1}\right] \quad (1)$$

where t_R and t_A are the solute retention time and the inert gas dead time, respectively, m_L is the mass of stationary liquid, F_A is the volumetric flow rate of the carrier gas measured at the column exit by a soap bubble flow meter at temperature T_{fm} and pressure P_{fm}, P_w is the vapor pressure of water at T_{fm}, and P_i and P_o are the column inlet and outlet pressures, respectively. According to the experimental apparatus used in this work, the pressure at the column exit is assumed to be equal to the pressure at which the soap bubble flow meter operates. The last term

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Table 1. Main Characteristics and Properties of Genosorb 300 and Genosorb 1843

	units	Genosorb 300 $CH_3(OCH_2CH_2)_nOCH_3$ $n = 3-8$	Genosorb 1843 $C_4H_9(OCH_2CH_2)_nOC_4H_9$ $n = 3-5$
appearance		dark	dark
molar mass	g/mol	280	270
boiling range (1013 mbar)	°C	>250	>270
cloud point	°C	−23	−44
vapor pressure (20 °C)	mbar	<0.002	$4 \cdot 10^{-4}$
density (20 °C)	g/cm^3	1.02−1.04	0.927
water absorption (20 °C)	wt %	∞	<3
solubility in water (20 °C)	wt %	∞	<2
viscosity (20 °C)	mm^2/s	7 − 8	4−5
specific heat (50 °C)	J/(g K)	2.13	2.13
thermal conductivity	W/(m K)	0.1693	0.1493
flash point	°C	137	154
ignition temperature	°C	210	205
stabilizer		2% stabilizer concentrate VP 1825	2% stabilizer concentrate VP 1852

in brackets in eq 1 takes into account the effect of the pressure drop across the column and, thus, the variations of density and velocity of the carrier gas.

The solute activity coefficient at infinite dilution γ_2^∞ is related to the specific retention volume by the following equation, which takes into account gas phase imperfections (Windsor and Young, 1967):

$$\ln \gamma_2^\infty = \ln \frac{273.15R}{M_L V_g^0 P_2^s} - \frac{(B_{22} - v_2^0)P_2^s}{RT_c} + \frac{2B_{2g} - v_2^\infty}{RT_c}(P_o J_3^4) \quad (2)$$

M_L is the molecular weight of the stationary liquid, R is the gas constant, P_2^s is the vapor pressure of the solute, T_c is the column absolute temperature, B_{22} is the second virial coefficient for the pure solute, B_{2g} is the cross-virial coefficient between solute and carrier gas, v_2^0 is the molar volume of the pure liquid solute, v_2^∞ is the partial molar volume of the solute at infinite dilution in the stationary phase, and J_3^4 is the carrier gas compressibility factor defined by Windsor and Young (1967) as

$$J_3^4 = \frac{3}{4}\left[\frac{(P_i/P_o)^4 - 1}{(P_i/P_o)^3 - 1}\right] \quad (3)$$

When helium is used as carrier gas, eq 2 can be approximated by (Martire et al., 1979; Nikolic et al., 1994)

$$\ln \gamma_2^\infty = \ln \frac{273.15R}{M_L V_g^0 P_2^s} - \frac{B_{22}P_2^s}{RT_c} \quad (4)$$

Partial molar excess enthalpy at infinite dilution $H_2^{E\infty}$ can be evaluated by temperature dependence of the activity coefficient at infinite dilution with the help of the Gibbs−Helmholtz relation:

$$\left(\frac{\partial \ln \gamma_2^\infty}{\partial\left(\frac{1}{T}\right)}\right)_{P,x} = \frac{H_2^{E\infty}}{R} \quad (5)$$

Experimental Section

The measurements were carried out using a Carlo Erba gas chromatograph, Model 2357, appropriately adapted. The thermal conductivity detector was interfaced to the data acquisition and integration system Alltech-RSL Chromlab III. The flow rate of the carrier gas, helium (>99.99%) in this work, was measured by a soap bubble flow meter positioned at the end of the chromatographic line and recording the pressure and temperature values at the flow meter. The column temperature was controlled within ±0.1 K. The pressure drop across the column, lying between 0.1 and 0.38 bar, was measured by a mercury differential manometer. The solid support used for the preparation of each column was an acid-washed diatomaceous earth, Chromosorb P-AW-DMCS 60−80 mesh, manufactured by Manville International Corp. The coating of the predried solid support with the partitioning liquid was carried out with a suitable solubilizer (dichloromethane in this work) in a rotary evaporator. The amount of solution (solubilizer plus stationary liquid) necessary for a complete wetting of the solid support was experimentally found to be about 240 mL/100 g of Chromosorb. After removal of the dichloromethane, the columns were carefully filled with the stationary phase. Stainless steel columns, inner diameter 5 mm, length 0.5−1.3 m, were prepared.

Considerations leading to eq 4 assume that a linear section of the sorption isotherm is involved and the only mechanism of retention is the dissolution of the solute vapor in the bulk stationary liquid. The first assumption is satisfied by injecting sufficiently small amounts of the solute sample in the carrier gas flow: 0.1−0.5 μL is suggested as a suitable volume (Conder and Young, 1978; Laub and Pecsok, 1978).

In this work liquid loadings of about 17% (w/w) and sample volumes of the injected solute between 0.2 and 0.3 μL were used. For these operating conditions and for the tested systems, the possible effect of adsorption on the solid support and at the gas−liquid interface can be neglected, as shown by Schiller and Gmehling (1992).

Results

The following organic high-boiling liquids belonging to the family of the glycol ethers and commercialized by Hoechst were examined: Genosorb 300 (mixture of polyethylene glycol dimethyl ethers, $CH_3(OCH_2CH_2)_nCH_3$ where $n = 3-8$) and Genosorb 1843 (mixture of polyethylene glycol dibutyl ethers, $C_4H_9(OCH_2CH_2)_nOC_4H_9$ where $n = 3-5$). The main characteristics and properties of these two liquid are listed in Table 1 (Loesemittel Hoechst AG, 1992).

The experimental values of the specific retention volumes V_g^0 at five different temperatures (42, 50, 62, 70, 90 °C) in the two liquids for the substances listed are collected in Tables 2 and 3. The solute activity coefficients at infinite dilution for the same experimental temperatures obtained by using eq 4 are listed in Tables 4 and 5. Except for the system tetrachloroethylene/Genosorb300, negative deviations from Raoult's law are obtained; this behavior is particularly desirable in the absorption step.

Table 2. Experimental Specific Retention Volume, V_g^0, for the Solvent Genosorb 300 as a Function of Temperature

	$V_g^0/(\mathrm{cm^3 \cdot g^{-1}})$				
solute	t = 42 °C	t = 50 °C	t = 62 °C	t = 70 °C	t = 90 °C
dichloromethane	427.9	295.8	195.8	139.5	69.35
trichloromethane	1053	684.7	381.8	283.1	138.5
tetrachloromethane	361	264.9	177.4	131.9	68.31
1,2-dichloroethane	1353	893.2	513.8	380.1	176.3
1,1,1-trichloroethane	380.7	277.8	187.3	142	72.99
trichloroethylene	911.2	599.9	334.2	254.8	128.8
tetrachloroethylene	1205	811.6	496.4	338.5	176.8
ethyl acetate	385.2	284.8	177.5	132.6	68.18
1,1,2-trichlorotrifluoroethane	52.45	44.86	30.54	24.53	14.35

Table 3. Experimental Specific Retention Volume, V_g^0, for the Solvent Genosorb 1843 as a Function of Temperature

	$V_g^0/(\mathrm{cm^3 \cdot g^{-1}})$				
solute	t = 42 °C	t = 50 °C	t = 62 °C	t = 70 °C	t = 90 °C
dichloromethane	281.1	203.1	144	105.6	55.4
trichloromethane	901	548.4	354.3	255.9	124.1
tetrachloromethane	414.9	299.4	213	166.7	87
1,2-dichloroethane	851	584.3	384.7	282	137.7
1,1,1-trichloroethane	400.2	291.9	204	154.5	84.8
trichloroethylene	1044	680.3	441.4	299.1	147.1
tetrachloroethylene	1911	1313	723.1	524.2	250.3
ethyl acetate	318.3	236.7	167.3	127.1	64.79
1,1,2-trichlorotrifluoroethane	95.70	72.28	47.26	37.92	21.66

Table 4. Experimental Activity Coefficients at Infinite Dilution, γ_2^∞, for the Solvent Genosorb 300 as a Function of Temperature

	γ_2^∞				
solute	t = 42 °C	t = 50 °C	t = 62 °C	t = 70 °C	t = 90 °C
dichloromethane	0.181	0.201	0.209	0.234	0.279
trichloromethane	0.152	0.176	0.211	0.222	0.260
tetrachloromethane	0.741	0.749	0.740	0.771	0.836
1,2-dichloroethane	0.268	0.294	0.326	0.333	0.383
1,1,1-trichloroethane	0.650	0.661	0.649	0.663	0.726
trichloroethylene	0.452	0.495	0.566	0.561	0.592
tetrachloroethylene	1.186	1.223	1.271	1.285	1.210
ethyl acetate	0.786	0.768	0.785	0.796	0.828

Table 5. Experimental Activity Coefficients at Infinite Dilution, γ_2^∞, for the Solvent Genosorb 1843 as a Function of Temperature

	γ_2^∞				
solute	t = 42 °C	t = 50 °C	t = 62 °C	t = 70 °C	t = 90 °C
dichloromethane	0.286	0.303	0.295	0.320	0.363
trichloromethane	0.184	0.227	0.236	0.255	0.301
tetrachloromethane	0.668	0.687	0.639	0.633	0.681
1,2-dichloroethane	0.441	0.466	0.451	0.466	0.508
1,1,1-trichloroethane	0.641	0.652	0.618	0.632	0.648
trichloroethylene	0.409	0.420	0.477	0.496	0.538
tetrachloroethylene	0.775	0.783	0.855	0.860	0.881
ethyl acetate	0.987	0.958	0.864	0.861	0.903

Vapor pressures for the solute were calculated from the four-parameter Wagner equation using constants reported by Reid et al. (1987). Comparison of this equation to other vapor pressure equations is reported in the literature, showing that the Wagner equation is the most accurate in various situations (Reid et al., 1987; McGarry, 1983). The second virial coefficients B_{22} were calculated by using a five-parameter correlation reported by Daubert and Danner (1985) using constants taken from the same compilation. The correlation gives an average error less than 5% for all of the tested substances, except for trichloroethylene and tetrachloroethylene for which errors of less than 25% and 50%, respectively, are estimated. Due to the lack of data, the second virial coefficient of 1,1,2-trichlorotrifluoroethane could not be computed.

In several works (Weidlich and Gmehling, 1987; Laub et al., 1978; Nikolic et al., 1994), it is shown that the dominant error in the calculation of the retention volume V_g^0 is the uncertainty regarding the mass of stationary phase in the column, m_L, and that the resulting error in the activity coefficient at infinite dilution γ_2^∞ takes into account the uncertainties in the vapor pressure P_2^s and in the second virial coefficient B_{22}. The error in the determination of V_g^0 is estimated to be less than 1% for the presented systems.

Figures 1 and 2 report the experimental γ_2^∞ values as a function of temperature and the interpolating lines, obtained with the least-squares method, for some of the examined solvents in both absorbing liquids (the root mean square percentage error between interpolated and experimental γ_2^∞ values is less than 4%). Analysis of Figures 1 and 2 suggests that a linear dependence of $\ln(\gamma_2^\infty)$ versus the reciprocal of the temperature may be assumed. With this assumption, eq 5 implies a constant value of the partial molar excess enthalpy at infinite dilution for the considered systems and in the utilized temperature range. The values of $H_2^{E\infty}$ obtained by the slope of the relative interpolating lines are listed in Table 6.

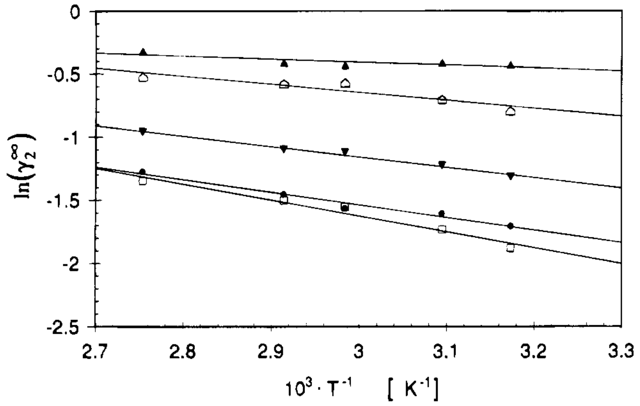

Figure 1. Experimental activity coefficients at infinite dilution, γ_2^∞, in the scrubbing liquid Genosorb 300 as a function of temperature: (●) dichloromethane; (□) trichloromethane; (▼) 1,2-dichloroethane; (▲) 1,1,1-trichloroethane; (○) trichloroethylene.

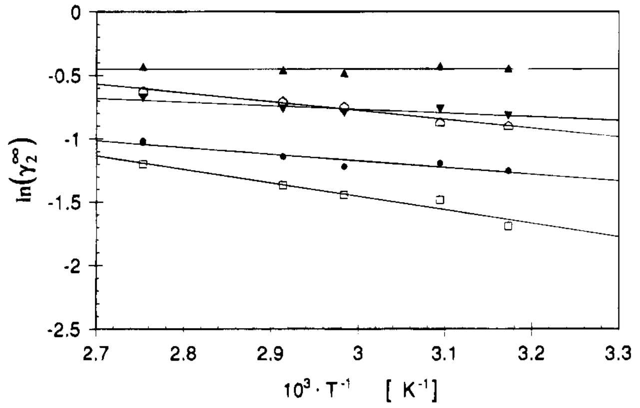

Figure 2. Experimental activity coefficients at infinite dilution, γ_2^∞, in the scrubbing liquid Genosorb 1843 as a function of temperature: (●) dichloromethane; (□) trichloromethane; (▼) 1,2-dichloroethane; (▲) 1,1,1-trichloroethane; (○) trichloroethylene.

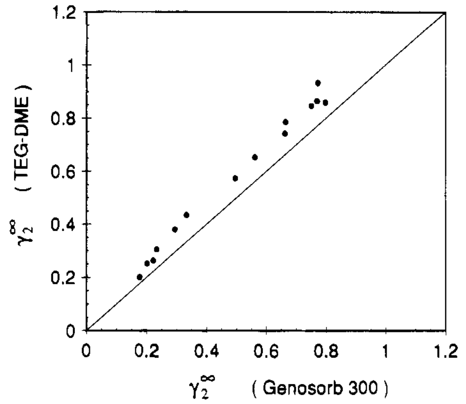

Figure 3. Parity plot of experimental γ_2^∞ values, at 50 and 70 °C, for Genosorb 300 and TEG-DME.

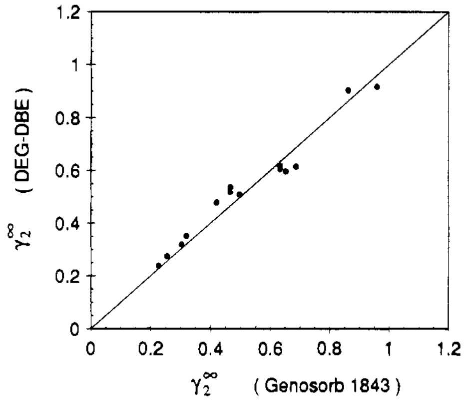

Figure 4. Parity plot of experimental γ_2^∞ values, at 50 and 70 °C, for Genosorb 1843 and DEG-DBE.

Table 6. Partial Molar Excess Enthalpy[a] at Infinite Dilution, $H_2^{E\infty}$, for the Solvents Genosorb 300 and Genosorb 1843

	$H_2^{E\infty}$/kJ·mol^{-1}	
solute	Genosorb 300	Genosorb 1843
dichloromethane	−8.0	−4.4
trichloromethane	−10.5	−8.9
tetrachloromethane	−2.3	0.2
1,2-dichloroethane	−6.8	−2.4
1,1,1-trichloroethane	−1.6	0.0
trichloroethylene	−5.3	−5.8
tetrachloroethylene	−0.5	−2.8
ethyl acetate	−1.2	2.1

[a] For temperature range see Tables 4 and 5.

Discussion

The activity coefficient at infinite dilution depends on the interactions existing in the ideal case in which a solute molecule is completely surrounded by solvent molecules. Different intensities of these interactions are correlated to the values of γ_2^∞ of the examined substances in a given absorbing liquid. Genosorb 300 and Genosorb 1843 are mixtures of polyethylene glycol dimethyl ethers (PEG-DME) and polyethylene glycol dibutyl ethers (PEG-DGE), respectively. Different intensities of the interactions can be explained by the formation of hydrogen bonds involving oxygen atoms of the washing liquids and hydrogen atoms, differently polarized due to the electron-attracting halogen atom, of the solvents, as suggested in the works of Zellhoefer et al. (1938a,b) on the solubility of various volatile halogenated compounds in different liquids.

Schiller and Gmehling (1992) report the experimental γ_2^∞ values of the substances considered in this study in five pure liquids among which are tetraethylene glycol dimethyl ether (TEG-DME), diethylene glycol dibutyl ether (DEG-DBE), and triethylene glycol dibutyl ether (TrEG-DBE). It can be observed that in glycol ethers the solubilities at infinite dilution are influenced not only by the type and number of monomers but also by the length of the alkyl chain.

Genosorb 300 is a mixture of PEG-DME (n = 3−8); the major percentage is represented by polymers with four, five, and six monomers (ethylene oxide). Activity coefficients at infinite dilution, at 50 and 70 °C, listed in Table 4, and the values reported in the work of Schiller and Gmehling (1992) are compared in Figure 3; better solubility of the examined substances in Genosorb 300 than in TEG-DME is shown. This behavior may be justified by the longer chain of the molecules of Genosorb 300 as compared with that of TEG-DME.

With regard to Genosorb 1843, the manufacturing firm does not provide any data on the composition of the mixture. However, the parity plot in Figure 4 shows good agreement of the values in DEG-DBE obtained by Schiller and Gmehling (1992) with those in Genosorb 1843 (the reported values are listed in Tables 5 and refer to two temperatures: 50 and 70 °C).

Conclusions

Gas−liquid chromatography allows fast, accurate determination of the solubility data at infinite dilution, necessary both for the selection of the most suitable scrubbing liquid and for the design of the absorption equipment.

Equilibrium data for the examined solvents are reported in the literature for pure scrubbing liquids of the alkylene glycol dialkyl ether type, while commercial absorption liquids are mixtures of glycol ethers at different grades of polymerization.

In this work two products manufactured by Hoechst were considered (Genosorb 300 and Genosorb 1843). A comparison of the experimental data produced in this work with those taken from the literature showed that activity coefficients at infinite dilution measured in the pure liquid, DEG-DBE, can be transferred to the homologue mixture, Genosorb 1843. On the contrary, the values obtained in the pure liquid, TEG-DME, if transferred to the mixture Genosorb 300 would induce an underestimation on the solubility data measured in the commercial product.

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