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# Activity Coefficients for Cyclohexane, Cyclohexene, and Benzene in Extractive Distillation Solvents Using Non-Steady-State Gas Chromatography

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The infinite-dilution activity coefficients of cyclohexane, cyclohexene, and benzene in four solvents, isophorone (CAS 78-59-1), dimethyl sulfoxide (CAS 67-68-5), dimethyl succinate (CAS 106-65-0), and propylene glycol (CAS 57-55-6), have been determined at temperatures ranging from 40 °C to 80 °C, by non-steady-state gas chromatography. These solvents are used commercially in the separation of mixtures of these hydrocarbons by extractive distillation, so infinite-dilution selectivities have been calculated and studied in relation to this separation. The experimental activity coefficients were compared with those calculated by the UNIFAC method.

## Introduction

Extractive and azeotropic distillation are used for the separation of close-boiling and azeotropic mixtures, respectively. The separation normally requires the addition of a solvent known as an entrainer. In extractive distillation the entrainer is a high-boiling-point solvent fed to the distillation column in order to modify the activity coefficients of the components to be separated, and hence the relative volatility. Solvent selection for extractive distillation cannot be made, in general, from pure component properties due to the large deviations of the mixtures from ideality.

At low or moderate pressures the relative volatility of two components, *i* and *j*, in the presence of a solvent, $\alpha_{ij}^{s}$, is given by

$$\alpha_{ij}^{s} = \frac{(y_i^s/x_i^s)}{(y_j^s/x_j^s)} = \frac{\gamma_i^s p_i^\circ}{\gamma_j^s p_j^\circ} \tag{1}$$

where *y* and *x* are the mole fractions of the components in the vapor and liquid phases, respectively, $\gamma$ is the activity coefficient, $p°$ is the vapor pressure, subscripts correspond to components *i* and *j*, respectively, and the superscript s indicates in the presence of the solvent.

Since the ratio $p_i^\circ/p_i^\circ$ is basically constant for small temperature changes, the influence of the solvent is usually quantified in terms of the so-called selectivity or separation factor, $S_{ij}$, defined as the ratio of the activity coefficients of the two key components in the presence of the solvent:

$$S_{ij} = \frac{\gamma_i^s}{\gamma_j^s} \tag{2}$$

As the activity coefficients depend on the phase composition and the effect of the solvent tends to increase as its concentration increases, it is a common practice to consider, at least in a preliminary solvent selection, the situation at infinite dilution. The selectivity at infinite dilution, $S_{ij}^\infty$, is defined as the ratio of the activity coefficients of both components at infinite dilution in the solvent:

$$S_{ij}^\infty = \frac{\gamma_i^{s\infty}}{\gamma_j^{s\infty}} \tag{3}$$

where $\gamma_i^{s\infty}$ and $\gamma_j^{s\infty}$ are the activity coefficients at infinite dilution of components *i* and *j* in the solvent, respectively.

A number of methods have been reported in the literature for the experimental determination of activity coefficients. A standard procedure is differential ebulliometry, in which the boiling point difference between a solution and the solvent is measured as a function of composition. It is a reliable but time-consuming technique. Gas−liquid chromatography (GLC) has been used extensively to obtain the activity coefficients at infinite dilution,[1−6] but a limitation of this technique is that the liquid phase (solvent) must have a low volatility in order to avoid the entrainment of the stationary phase. This problem can be overcome, at least to some extent, by a combination of GLC and liquid−liquid chromatography (LLC), using a high-molecular-weight compound as the liquid phase in both techniques.[7,8] Vega and Coca[9] have recently reviewed these chromatographic techniques.

Another method is non-steady-state gas chromatography (NSGC), in which the solvent is itself being eluted from the column during the chromatographic experiment. NSGC has been used to determine activity coefficients of solutes in volatile solvents.[10−14]

Benzene, cyclohexane, and cyclohexene are hydrocarbons present in different streams in the petrochemical industry, for example in the products of partial catalytic hydrogenation of benzene. Because of their close boiling points (benzene, 80.10 °C; cyclohexane, 80.74 °C; cyclohexene, 82.98 °C) extractive distillation is a process potentially suitable for the separation of these hydrocarbon mixtures. Several references on the separation of cyclohexane, cyclohexene, and benzene by extractive distillation can be found, mainly in the patent literature, because of its industrial importance. Some of the commercial extractive solvents suitable for this separation are sulfones,[15−17]

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morpholines,[18,19] amides,[20,21] esters,[22,23] and pyrrolidones.[24−26] However, available data do not allow comparison of the solvents' performance.

The present investigation was undertaken to assess several solvents for the separation by extractive distillation of cyclohexane, cyclohexene, and benzene mixtures, using the NSGC technique to determine activity coefficients at infinite dilution. The four solvents studied in this work were as follows: isophorone, ISOP (CAS 78-59-1); dimethyl sulfoxide, DMSO (CAS 67-68-5); dimethyl succinate, DMS (CAS 106-65-0); and propylene glycol, PG (CAS 57-55-6).

## Theoretical Background

In NSGC, the column is either initially packed with a GC uncoated solid support or is an uncoated fused silica capillary column. The liquid solvent is injected into the heated injection port of a gas chromatograph. The vapors produced are transported into the column by the carrier gas, and if the column temperature is lower than the solvent boiling point, the solvent condenses in a more or less uniform film on the support or onto the walls of the capillary column. Once the solvent has equilibrated in the column and the excess vapors have been eluted, the gas chromatograph detector stabilizes and gives a high, flat baseline (plateau) corresponding to the eluting solvent vapors. As the carrier gas flows through the column, it becomes saturated with solvent vapor. Solvent bleeds out of the column at a uniform, continuous rate. Once a steady baseline is obtained, small amounts of a solute, more volatile than the solvent, are injected at small time intervals. As the total mass of solvent in the column decreases with time, the retention time of an injected solute decreases over the lifetime of the column.

An analysis of the system[10] leads to a simple equation relating the infinite dilution activity coefficient of the solute ($i$) in the volatile solvent (s), $\gamma_{\mathrm{i}}^{\mathrm{s}\infty}$, to the ratio of their vapor pressures, and the rate at which retention time decreases with time of injection.

Assuming ideal behavior of the vapor phase and that the solutions are sufficiently diluted so that Henry's law applies, the solute net retention volume, $V_{\mathrm{N}}$, in steady-state (conventional) gas chromatography is related to the limiting activity coefficient by the expression

$$V_{\mathrm{N}} = K_i V_{\mathrm{s}} = RTn_{\mathrm{s}}/p_i^{\circ}\gamma_i^{\mathrm{s}\infty} \tag{4}$$

where $K_i$ and $p_i^{\circ}$ are the distribution coefficient and vapor pressure of the solute, $V_{\mathrm{s}}$ and $n_{\mathrm{s}}$ are the volume and the number of moles of solvent in the column, and $T$ is the column temperature.

In NSGC, the decrease in retention volume is proportional to the decrease in the amount of solvent:[12]

$$\Delta V_{\mathrm{N}} = K_i \Delta V_{\mathrm{s}} = (RT/p_i^{\circ}\gamma_i^{\mathrm{s}\infty})\Delta n_{\mathrm{s}} \tag{5}$$

At constant carrier gas flow rate, $F$, and column temperature, the loss of solvent is directly proportional to time. Over a time interval $\Delta t = t_1 - t_2$, where $t_1$ and $t_2$ are two different times of injections of solute, the loss of solvent is

$$(\Delta n_{\mathrm{s}}/\Delta t) = n_{\mathrm{v}}F = p_{\mathrm{s}}^{\circ}F/RT \tag{6}$$

where $n_{\mathrm{v}}$ is the number of moles of volatilized solvent per unit volume of gas phase. Combining eqs 4−6 and taking into account that for a constant carrier gas flow rate $\Delta V_{\mathrm{N}}/F = \Delta t_{\mathrm{R}}$:

$$\gamma_i^{\mathrm{s}\infty} = -\left(\frac{p_{\mathrm{s}}^{\circ}}{p_i^{\circ}}\right)\Big/\left(\frac{\Delta t_{\mathrm{R}}}{\Delta t}\right) \tag{7}$$

Equation 7 indicates that the limiting activity coefficients can be determined if the vapor pressures and the solute retention times measured at different injections are known. Although, in principle, only two injections are needed, the larger the number of solute injections made, the better the accuracy of the experimental data.

## Experimental Section

A Varian Star 3400 gas chromatograph equipped with a flame ionization detector and a septum-equipped programmable injector was used with a Supelco 30 m × 0.25 mm i.d. uncoated and deactivated fused silica capillary column. Measurements were made at column temperatures in the range (40−80) °C, using helium as carrier gas. Chromatographic data were collected in a Varian Star chromatography workstation.

To load the column with a uniform and relatively thick film of solvent, the column temperature was set to a value slightly higher than the solvent boiling point. About 10 $\mu$L of the liquid solvent was injected into the column. Condensation of the solvent onto the walls of the capillary column was achieved by decreasing slowly the column temperature to the desired operating value and using a very low carrier gas flow rate.

Once the baseline was stabilized, solutes were injected at different times, without waiting for the complete elution of the sample before making a new injection. The higher the number of solute injections made over the life period of the column, the better was the definition of the slope of the graph of retention time versus injection time. The complete evaporation of the solvent was detected by a sharp drop in the baseline, which indicates the need for a new injection of fresh solvent.

Solutes (benzene, cyclohexane, and cyclohexene) were reagent grade or HPLC grade from Aldrich (Aldrich Chemie AG, Steinheim, Germany) or Fluka (Fluka Chemie AG, Buchs, Switzerland) with a minimum purity of 99.5%. The solvents used (isophorone, dimethyl sulfoxide, dimethyl succinate, and propylene glycol) were also reagent grade from Aldrich or Riedel−de Haën (Riedel−de Haën AG, Seelze, Germany) with a minimum purity of 99.0%.

## Results and Discussion

The infinite-dilution activity coefficient, $\gamma_{\mathrm{i}}^{\mathrm{s}\infty}$, can be determined from the slope of a plot of retention time versus injection time, and the solute/solvent vapor pressure ratio, according to eq 7.

Vapor pressures for isophorone, dimethyl sulfoxide, and dimethyl succinate were taken from *TRC Data Base*,[27] and those for propylene glycol were calculated from experimental data taken from *Solvents Guide*[28] and fitted to the Antoine equation. Vapor pressures for benzene were taken from Gmehling et al.,[29] and those for cyclohexane and cyclohexene were taken from Yaws.[30]

To test the experimental technique, the activity coefficient of $n$-hexane at infinite dilution in toluene at 40 °C was determined. The value of 1.64 obtained is in good agreement with the value of 1.62 reported by Landau et al.[12] We have fitted the vapor−liquid equilibrium data of hexane and toluene, taken from the *TRC Data Base*,[31] to the NRTL equation (with an average deviation of 0.982%). Using the NRTL equation, the activity coefficient of hexane

**Table 1. Activity Coefficients at Infinite Dilution for Cyclohexane, Cyclohexene, and Benzene in Several Solvents at Different Temperatures**

| | | activity coefficient at infinite dilution[a] | | | |
|---|---|---|---|---|---|
| solute | $t$/°C | ISOP | DMSO | DMS | PG |
| benzene | 40 | 1.40 ± 0.04 | | | |
| | 55 | 1.78 ± 0.26 | 3.11 ± 0.08 | 2.36 ± 0.10 | |
| | 70 | 1.25 ± 0.02 | 3.02 ± 0.05 | 1.78 ± 0.05 | 12.50 ± 1.05 |
| | 80 | 0.99 ± 0.13 | 2.67 ± 0.04 | 1.52 ± 0.03 | 9.38 ± 0.33 |
| | 90 | | | 1.36 ± 0.12 | 7.96 ± 0.38 |
| cyclohexene | 40 | 2.47 ± 0.07 | | | |
| | 55 | 2.78 ± 0.13 | 10.94 ± 0.36 | 5.16 ± 0.17 | |
| | 70 | 2.08 ± 0.03 | 9.53 ± 0.19 | 4.05 ± 0.09 | 31.93 ± 7.36 |
| | 80 | 2.51 ± 0.05 | 8.14 ± 0.35 | 3.53 ± 0.12 | 18.45 ± 0.62 |
| | 90 | | | | 11.50 ± 1.15 |
| cyclohexane | 40 | 4.18 ± 0.27 | | | |
| | 55 | 3.38 ± 0.11 | 23.70 ± 3.45 | 7.40 ± 0.26 | |
| | 70 | 2.68 ± 0.05 | 19.34 ± 0.57 | 5.88 ± 0.21 | 48.13 ± 4.61 |
| | 80 | 2.92 ± 0.09 | 16.02 ± 1.51 | 5.27 ± 0.33 | 29.62 ± 1.75 |
| | 90 | | | | 18.09 ± 3.35 |

[a] ISOP = isophorone; DMS = dimethyl succinate; DMSO = dimethyl sulfoxide; PG = propylene glycol.

**Table 2. Estimated UNIFAC Activity Coefficients at Infinite Dilution for Cyclohexane, Cyclohexene, and Benzene in Several Solvents at Different Temperatures**

| | | activity coefficient at infinite dilution[a] | | | |
|---|---|---|---|---|---|
| solute | $t$/°C | ISOP | DMSO | DMS | PG |
| benzene | 40 | 0.85 | 2.91 | 1.08 | 6.89 |
| | 55 | 0.84 | 2.80 | 1.04 | 6.64 |
| | 70 | 0.84 | 2.71 | 1.00 | 6.40 |
| | 80 | 0.83 | 2.66 | 0.98 | 6.24 |
| | 90 | 0.82 | 2.60 | 0.96 | 6.08 |
| cyclohexene | 40 | 1.21 | 11.15 | 2.58 | 12.76 |
| | 55 | 1.19 | 9.88 | 2.48 | 13.00 |
| | 70 | 1.17 | 8.86 | 2.38 | 12.94 |
| | 80 | 1.15 | 8.28 | 2.31 | 12.77 |
| | 90 | 1.14 | 7.77 | 2.23 | 12.51 |
| cyclohexane | 40 | 1.44 | 17.78 | 3.41 | 14.50 |
| | 55 | 1.41 | 15.52 | 3.06 | 13.69 |
| | 70 | 1.38 | 13.72 | 2.76 | 12.92 |
| | 80 | 1.36 | 12.71 | 2.58 | 12.42 |
| | 90 | 1.35 | 11.82 | 2.42 | 11.94 |

[a] ISOP = isophorone; DMS = dimethyl succinate; DMSO = dimethyl sulfoxide; PG = propylene glycol.

**Table 3. Selectivities at Infinite Dilution for Cyclohexane, Cyclohexene, and Benzene in Several Solvents at Different Temperatures**

| | | infinite-dilution selectivities in solvent:[a] | | | |
|---|---|---|---|---|---|
| solute | $t$/°C | ISOP | DMSO | DMS | PG |
| cyclohexane + benzene | 40 | 2.98 | | | |
| | 55 | 1.90 | 7.62 | 3.13 | |
| | 70 | 2.14 | 6.40 | 3.31 | 3.85 |
| | 80 | 2.96 | 6.01 | 3.46 | 3.16 |
| | 90 | | | | 2.27 |
| cyclohexene + benzene | 40 | 1.76 | | | |
| | 55 | 1.56 | 3.52 | 2.19 | |
| | 70 | 1.66 | 3.15 | 2.28 | 2.55 |
| | 80 | 2.55 | 3.06 | 2.31 | 1.97 |
| | 90 | | | | 1.45 |
| cyclohexane + cyclohexene | 40 | 1.69 | | | |
| | 55 | 1.22 | 2.17 | 1.43 | |
| | 70 | 1.29 | 2.03 | 1.45 | 1.51 |
| | 80 | 1.16 | 1.97 | 1.49 | 1.61 |
| | 90 | | | | 1.57 |

[a] ISOP = isophorone; DMS = dimethyl succinate; DMSO =dimethyl sulfoxide; PG = propylene glycol.

at infinite dilution in toluene was calculated. The value obtained, 1.66, also agrees very well with our NSGC data.

Activity coefficients at infinite dilution obtained in this work for cyclohexane, cyclohexene, and benzene as solutes in the solvents tested at different temperatures, together with the corresponding 95% confidence intervals, are listed in Table 1.

Results in Table 1 show that, for all the solvents tested, the activity coefficients at infinite dilution, at a given temperature, increase in the order benzene, cyclohexene, cyclohexane, which corresponds to decreasing polarity and increasing molar volume. The activity coefficients at infinite dilution decrease as temperature increases for all solute−solvent pairs.

Activity coefficients at infinite dilution can be estimated by the two-parameter (temperature-dependent parameters) Original UNIFAC method. The values of these coefficients calculated by using the GAMMA program of the IVC-SEP package[32] are given in Table 2. The activity coefficients estimated by the UNIFAC method show large deviations with respect to those determined experimentally. Similar disagreement between experimental and UNIFAC estimated activity coefficients at infinite dilution was also found for hydrocarbons in vegetable oils.[33] Deviations are likely due to the fact that the mathematical form of the UNIFAC equation sometimes cannot reproduce the observed steep change of the activity coefficients with composition as the limiting activity coefficient region is approached, especially when parameters are based on activity coefficient data remote from the dilute region.

Infinite-dilution selectivities have been calculated using eq 3 (Table 3). Considering selectivity, the best solvents for the cyclohexane−benzene separation are (in decreasing order) DMSO, PG, DMS, and ISOP with selectivities in the range 6.4 to 2.14. The trends for cyclohexene−benzene and cyclohexane−cyclohexene separations are the same, but selectivities are lower. According to these results, DMSO is the best solvent for the benzene, cyclohexene, and cyclohexane separation, as it presents the highest values of selectivities. Results indicate that the separation of cyclohexane−cyclohexene is the most difficult, despite the difference of boiling points (2.24 °C): much higher than those for cyclohexane−benzene (0.64 °C). This behavior has also been observed with other solvents.[14]

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