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Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Ecenarro, O. , Madariaga, J. A. , Navarro, J. , Santamaría, C. M. , Carrión, J. A. and Savirón, J. M.(1991) 'Direction of Separation and Dependence of Feed Concentration in Liquid Thermogravitational Columns', Separation Science and Technology, 26: 8, 1065 — 1076

To link to this Article: DOI: 10.1080/01496399108050514

URL: http://dx.doi.org/10.1080/01496399108050514

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SEPARATION SCIENCE AND TECHNOLOGY, 26(8), pp. 1065–1076, 1991

Direction of Separation and Dependence of Feed Concentration in Liquid Thermogravitational Columns

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Abstract

The direction of separation in a thermogravitational column was studied for 12 binary mixtures with the following components: Benzene, toluene, *n*-hexane, *n*-heptane, cyclohexane, carbon tetrachloride, tetrabromoethane, and tetrachloroethane. The separation sign is governed by the density of the components. A study was also made of the effect of feed concentration on the separation value. The shape of the curve relating separation and charge concentration was found to depend strongly on "molecular mobility" and molecular structure.

1. INTRODUCTION

On applying a temperature gradient to a binary mixture of liquids, in general a concentration gradient is set up in the bulk of the fluid, leading to partial separation of the mixture components. This phenomenon is known as the "Soret effect" or liquid phase thermal diffusion. However, the degree of separation obtained through the Soret effect is extremelly small. Today, in order to use thermal diffusion as a separation technique or for obtaining reliable values of the thermal diffusion factor, thermogravitational columns are used. In these, the convection becomes superimposed over the Soret effect, giving rise to an ascending flow of liquid on the hot wall and a descending flow on the cold wall. Owing to the Soret

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effect, these currents have different concentrations, and one thus obtains a net difference in the composition of the mixture at both ends of the column.

To be able to take advantage of this separation in industrial processes, it would be of great value to have *a priori* knowledge of which component is enriched in the top and bottom parts of the column, together with the degree of separation and the effect of the initial composition on it. For the case of mixtures of gases, the separation sign is well known and, according to kinetic theory, is given by the relative masses of the molecules participating. The component with the lower mass is enriched at the top part of the column. However, the situation is much more complex for mixtures of liquids. Since there is no correct kinetic theory available, the number of empirical rules attempting to predict which component will gather on the hot or cold wall is very large. In this sense, Tyrrell (*1*) concluded that the parameter governing the direction of separation is the vaporization heat per unit of volume. In principle, this quantity determines heat transference and hence the direction in which the components in the thermal gradient will migrate. Intimately linked with this is the solubility parameter, δ, which is no more than the square root of the previous vaporization heat. According to this rule, the component with the highest vaporization heat or solubility parameter will tend to gather on the cold wall, and hence, if there are no problems brought about by the so-called "forgotten effect," the mixture will be enriched in this component at the lower end of the column. The literature also cites other, simpler norms that aim at determining the direction of separation. An example is the work of Bott and Whysall (*2*), who indicate that the component accumulating on the cold wall will be the one with the highest boiling point and molecular weight. However, none of the above rules can be taken as indicators of the direction of separation because exceptions to the rules are too common. In the light of this, and in agreement with Tyrrell (*1*), it seems appropriate to have available a large number of reliable experimental data of the separation sign, for a wide range of mixtures, obtained by using the same experimental technique.

In the present work we have determined separation in a thermogravitational column without reservoirs for 12 binary mixtures with different compositions. The components of such mixtures (benzene, toluene, *n*-hexane, *n*-heptane, cyclohexane, carbon tetrachloride, tetrabromoethane, and tetrachloroethane) have molecules with very different molecular structures: rings, chains, and tetrahedra, with no association or formation of complexes among them. We have also analyzed the effect of the initial feed composition on the final separation value in a thermal diffusion column.

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2. EXPERIMENTAL

The thermal diffusion column for liquids employed in the present work was the same as that used in a previous set of experiments (*3*). The column essentially consists of two concentric stainless steel tubes of the following dimensions: Length (L) = 0.9 m, external diameter of internal tube ($2r_2$) = 59 mm, and internal diameter of external tube ($2r_1$) = 60.9 mm. These were kept at different temperatures by circulating water from two thermostatted baths. The separation between the cylinders, or gap, is 0.95 mm. In most cases, operating temperatures were as follows: Cold wall (T_C) = 309.0 K and hot wall (T_H) = 313.0 K, the mean temperature of the liquid being the arithmetic mean of both: T_m = 311.0 K.

The mixtures were prepared volumetrically from pure reagent-grade components. Handling of mixtures was standard for these cases and has been reported in the literature (*3*).

Determination of mass concentration was carried out with a Zeiss refractometer, with a nominal precision of 2×10^{-5} in the measurement of the refraction index.

The separation factor is defined as usual:

$$q = \frac{x_T(1 - x_B)}{x_B(1 - x_T)}$$

where x_T and x_B are the molar fractions of the less dense component at the top and the bottom of the column, respectively, and were obtained at steady-state. Also, we define v_0 as the initial volume fraction of one component, with Δv being the separation volume fraction, that is, the difference of volume fractions between the column ends. In what follows we shall use this quantity as a criterion of separation for convenience instead of the above-defined separation factor. The reproductibility of the experimental separation data was estimated to be better than 5%.

3. RESULTS AND DISCUSSION

3.1. Direction of Separation

The results obtained for the separation sign in our column are shown in Table 1. The last column of the table shows which of the two components of the mixture is enriched at the bottom of the thermogravitational column. The table also shows the molecular masses (M_i), densities (ρ_i), boiling points (T^i_{bp}), and solubility parameters (δ_i) of component i of the binary systems. All these data were taken from the *Handbook of Chemistry and Physics* (*7*). From the results shown, it may be inferred that for these

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TABLE 1[a]

System (1)–(2)	M_1	M_2	ρ_1	ρ_2	δ^1	δ^2	T^1_{bp}	T^2_{bp}	Enriched component at lower end
B–nHX	78.12	86.12	0.878	0.660	9.2	7.3	80	69	B
B–nHP	78.12	100.21	0.878	0.683	9.2	7.4	80	98	B
B–CHX	78.12	84.16	0.878	0.779	9.2	8.2	80	81	—
T–CHX	92.15	84.16	0.867	0.779	8.9	8.2	111	81	—
T–nHX	92.15	86.12	0.867	0.660	8.9	7.3	111	69	T
T–nHP	92.15	100.21	0.867	0.683	8.9	7.4	111	98	T
CHX–nHX	84.16	86.12	0.779	0.660	8.2	7.3	81	79	CHX
CHX–nHP	84.16	100.21	0.779	0.683	8.2	7.4	81	98	CHX
TCC–nHX	153.82	86.12	1.594	0.660	8.6	7.3	77	69	TCC
TCC–CHX	153.82	84.16	1.594	0.779	8.6	8.2	77	81	TCC
TCC–nHP	153.82	100.21	1.594	0.683	8.6	7.4	77	98	TCC
TCC–B	153.82	78.12	1.594	0.878	8.6	9.2	77	80	TCC
TBE–TCE	346.00	168.00	2.966	1.595	10.7	9.4	—	146	TBE

[a] B = benzene; T = toluene; nHX = *n*-hexane; nHP = *n*-heptane; CHX = cyclohexane; TCC = carbon tetrachloride; TBE = tetrabromoethane; TCE = tetrachloroethane; M_i = molecular weight of component *i*; ρ_i = density of component *i*; δ^i = solubility parameter of component *i*; T^i_{bp} = boiling point temperature of component *i*.

simple binary systems—that is, without molecular association and of ideal or nearly ideal behavior—the empirical rules indicating that the component with the highest molecular mass and highest boiling point would migrate toward the bottom of the column are not valid. In this sense, note, for example, the benzene–*n*-heptane and cyclohexane–*n*-heptane systems. With respect to the solubility parameter, which (according to Tyrrell) would predict the behavior of the direction of separation, the only exception from among the systems studied by us is carbon tetrachloride and benzene, whose solubility parameters have values of 8.6 and 9.2 $(\mathrm{cal/cm^3})^{1/2}$, respectively. In any case, in the light of the results shown in Table 1, it may be deduced that the density of the components is the parameter of greatest interest for determining the direction of separation, the component with the highest density being the one that gathers at the bottom of the column. All the mixtures studied in the present work with non-null separation are governed by this simple rule.

This conclusion can be extended to the results reported by other authors concerning mixtures displaying more complex behavior than the ideal and in columns with reservoirs at the ends. Table 2 shows the results of Bott et al. for binary systems composed of quinoline, dodecane, *n*-heptane, propyl iodide, and xylenol. The density and solubility parameter govern the direction of separation.

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TABLE 2[a]

System (1)–(2)	M_1	M_2	ρ_1	ρ_2	δ^1	δ^2	Enriched component at lower end	Ref.
Q–nDDC	129.30	170.30	1.090	0.751	13.9	7.0	Q	4
PI–nHP	170.00	100.21	1.748	0.683	9.0	7.4	PI	4
XOL–nDDC	—	170.30	0.983	0.751	9.4	7.0	XOL	4
T–nHP	92.15	100.21	0.867	0.693	8.9	7.4	T	5
CHX–nHX	86.12	84.16	0.779	0.660	8.2	7.3	CHX	5
EB–nHP	106.17	100.21	0.867	0.683	7.8	7.4	EB	5
MCHX–nHP	98.19	100.21	0.769	0.683	7.8	7.4	MCHX	5
TCEN–nHP	165.83	100.21	1.623	0.683	9.2	7.4	TCEN	5
mX–iOC	106.17	114.23	0.864	0.692	8.8	6.9	mX	5
EB–nOC	106.17	114.23	0.867	0.702	8.8	7.6	EB	5
ACF–nNN	120.16	128.00	1.028	0.718	9.8	7.8	ACF	5
TCEN–POL	165.83	60.11	1.623	0.804	9.2	10.5	TCEN	5
TCEN–nBOL	165.83	74.12	1.623	0.810	9.2	10.6	TCEN	5
ALB–nHXOL	108.15	102.18	1.042	0.814	11.4	9.8	ALB	5
ALB–nOCOL	108.15	130.23	1.042	0.827	11.4	9.3	ALB	5
CHXOL–nHXOL	100.16	102.18	0.962	0.814	10.6	10.7	CHXOL	5
CLF–B	119.38	78.12	1.483	0.878	9.3	9.2	CLF	6
CLF–ACN	119.38	58.08	1.483	0.789	9.3	9.9	CLF	6
CLF–T	119.38	92.15	1.483	0.867	9.3	8.9	CLF	6
CLF–MST	119.38	120.20	1.483	0.865	9.3	8.8	CLF	6

[a] B = benzene; T = toluene; mX = *m*-xylene; EB = ethylbenzene; nHX = *n*-hexane; nHP = *n*-heptane; nOC = *n*-octane; iOC = isooctane; nNN = *n*-nonane; nDDC = *n*-dodecane; CHX = cyclohexane; MCHX = methylcyclohexane; POL = propanol; nBOL = *n*-butanol; nHXOL = *n*-hexanol; CHXOL = cyclohexanol; ALB = benzyl alcohol; XOL = xylenol; CLF = chloroform; Q = quinoline; TCEN = 1,1,2,2-tetrachlorethylene; MST = mesitylene; PI = propyl iodide; ACN = acetone; ACF = acetophenone; M_i = molecular weight component *i*; ρ_i = density component *i*; δ^i = solubility parameter component *i*.

Table 2 also shows the results obtained by Morgado (*5*) for systems in which, in principle, there could be chemical associations and which, in any case, are not quasi-ideal. For all the systems offered, density governs the migration of the components along the column. The solubility parameter is also a good indicator for predicting the separation sign, although in the case of the mixture of cyclohexanol–*n*-hexanol, with very similar solubility parameters, there is a net separation.

The results obtained by Ma and Beyerlein, shown at the bottom of Table 2, also seem to confirm the importance of density for accounting for the direction of separation. The rule of the solubility parameter, however, fails to predict the direction of separation in the chloroform–acetone system. It should be noted that the validity of the solubility parameter rule is not completely general since, according to Tyrrell, the theory leading to this is only valid for certain types of solution, so that exceptions to the rule are not uncommon.

Tyrrell (*1*) reported results on the direction of separation for a large number and types of binary mixtures. The data thus collected were taken from diverse sources and obtained by using different experimental techniques: thermogravitational columns, stirred diaphragm cells, and the pure Soret effect. The separation direction of practically all these mixtures is governed by the criterion proposed in the present work: the denser component is enriched at the bottom of the column. Furthermore, for mixtures whose components have similar densities and structures (i.e., benzene–cyclohexane and toluene–cyclohexane), the degree of separation is very small or null.

According to the proposed density criterion, in the benzene–cyclohexane and toluene–cyclohexane systems one would expect that benzene and toluene, respectively, would be the components that would become enriched at the bottom of the column. However, in our experimental measurements no separation at all was found. The measurements were made at different concentrations [$0.28 < x_0 < 0.76$ molar fractions of benzene (toluene)], under different working conditions ($298 < T_m < 320$ K), and operation times greater than 100 h. Using a thermogravitational column, Jones and Milberger (*8*) did not detect separation for the benzene–cyclohexane mixture either. This was also the case for Korsching et al. (*9*) and Van Ness (*10*) who used the same mixture. Similar results were reported by Debye and Bueche (*11*). According to Jones and Milberger (*8*), such behavior can be accounted for in terms of the abnormal property of the mixture, which would prevent the individual molecules of each component from being free to diffuse independently throughout the system. Moreover, in the case of these mixtures it should be noted that the geometric shape of the molecules is quite similar, and this could have very strong effects on the relative mobility of the molecules. In gases, the molecules are sufficiently distant from one another and the molecular configurations do not have important effects on mobility, so that the effect of mass is predominant in affecting separation. In organic liquid media, the molecules are sufficiently close to one another for the molecular configurations to affect individual movement.

In any case, the proposed rule of densities is of more general validity. Even the data of Prigogine (*12*), obtained with a parallel plate thermogravitational column with reservoirs at the ends, are governed by this rule if one discards the bromobenzene–carbon tetrachloride and tetrabromoethane–tetrachloroethane systems, for which an inversion in separation appears. These authors attribute this to the forgotten effect. In order to

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further elucidate such an anomaly, in this series of experiments we determined the separation and the time course of the tetrabromoethane–tetrachloroethane system with a view to analyzing the effect of density on the direction of separation or, in other words, whether any large difference in the densities of the components would lead to an inversion in the direction of separation. The experimental results in closed columns contrast with those of Prigogine, and again the component with the highest density and solubility parameter was found to be enriched at the bottom of the column, with no kind of inversion in the dependence of separation on time. The values of the logarithm of separation at steady-state after extrapolation to $\Delta T = 0$ K, according to Ref. *4*, were as follows: $\ln q_{\infty}^{0} = 0.173$ for $x_0 = 0.33$ and $\ln q_{\infty}^{0} = 0.167$ for $x_0 = 0.51$, where x_0 is the initial molar fraction of tetrabromoethane, the mean temperature being $T_m = 311.0$ K. The Saxton (*13*) measurements of the Soret coefficient carried out with diaphragm cells are in agreement with our own insofar that tetrachloroethane is the component that concentrates in the chamber at the higher temperature.

According to Tyrrell (*1*), the anomalous results obtained by Prigogine for these systems cannot be interpreted only in terms of the effect of density on the changes in concentration in the column. The presence of large reservoirs at the ends of the columns used by Prigogine may have affected the results on the direction of separation. In such reservoirs, changes in density may give rise to remixing currents that would affect the thermal diffusion in the column (*1*). Such currents become stronger with an increase in the relative differences between the densities of the components and are practically negligible for gaseous systems. If the thermogravitational columns employed—as in the present case—do not have reservoirs, the inversion phenomena due to density differences are absent and the results obtained are those expected according to the rules proposed for densities and solubility parameters.

To be explicit, from the present results it may be inferred that the density is the most important parameter when one wishes to discover which component of the binary mixture will become enriched at the bottom of the column. Nevertheless, the existence of a difference in densities between the components will not guarantee that the components will be separated or, when they are, the magnitude of this separation.

From the fact that the denser component is enriched at the bottom, one cannot deduce "*a priori*" the direction of thermal diffusion. In effect, if the denser component migrates to the cold wall, it is obviously enriched at the bottom of the column. But if this component migrates to the hot wall and the forgotten effect is important enough to produce reversion of the convective flow, it will also concentrate at the bottom. To decide the

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sign of the thermal diffusion factor, it is necessary to analyze the nonsteady behavior of the column operation. In the first case, a normal separation time evolution can be expected. On the contrary, in the second one, the time evolution is complicated because the forgotten effect causing the initial flow reversion vanishes when the separation process approaches steady-state. In this respect, we have shown (*15*) that steady-state is not compatible with a reversion of the convective flow.

In the mixtures we have considered, the approach to equilibrium does not present any anomaly, and, in fact, we have obtained correct values of the ordinary diffusion coeffient from nonsteady separation measurements in recent works (*16*, *17*). Therefore, we conclude that in these mixtures the denser component migrates to the cold wall.

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3.2. Effect of Initial Composition on Separation

The effect of the initial concentration of the mixture on steady separation has been studied by Debye and Bueche (*11*), De Groot (*14*), Jones and Milberger (*8*), and, more recently, by Bott and Whysall (*2*). These authors have suggested that from their results it is possible to deduce important conclusions that should allow one to describe the mechanism of thermal diffusion in liquids, which is still not fully understood.

For the ideal case, Tyrrell (*1*) has pointed out that the dependence of separation, Δv, on the initial composition can be described as a parabola with a maximum at $v_0 = 0.5$. In the present work we chose to work with the volume fraction in order to compare our own findings with the results of Jones and Milberger (*8*) and Bott and Whysall (*2*).

Figure 1 shows the results obtained by us for separation, Δv, against composition, v_0, of the initial mixture in a benzene–*n*-heptane system for a working mean temperature of 311.0 K and a temperature difference between the cylinder walls of 23 K. It may be seen that the maximum separation does not occur for an initial equivolumetric mixture of benzene–*n*-heptane but rather takes place for an initial concentration of 60% by volume of benzene. Almost identical results were obtained by Bott and Whysall (*2*) in their column. Also, like these authors, we observed a difference in separations when the column is fed with a mixture rich in benzene or *n*-heptane. Thus, for a mixture with an initial composition of around 15% and another with 85%, both in benzene, the separation values reached in the latter case proved to be double that of the benzene-poor initial mixture. In other words, there seems to be "difficulty" in separating *n*-heptane-rich mixtures.

When the mixture studied is benzene–*n*-hexane, under the same experimental conditions, the results obtained concerning the position of the maximum are similar to those observed in the previous case (Fig. 1).

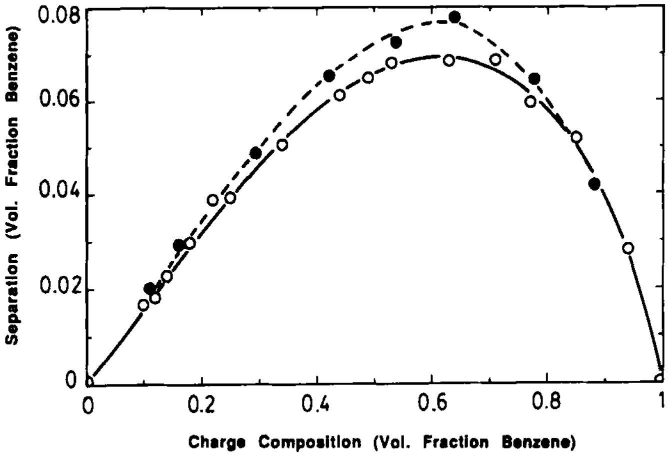

FIG. 1. Separation vs initial composition for (○) benzene–*n*-heptane and (●) benzene–*n*-hexane systems. The solid line is the best cubic fit to the experimental data.

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However, a striking feature is the effect of the chain-shaped molecule—in this case, *n*-hexane—leading the separation to increase compared with the previous system containing *n*-heptane.

For these systems, as may be seen in Fig. 1, the parabolic relationship between separation and initial concentration loses all relevance. The asymmetry of these curves is due—according to Bott—to the great mobility of systems rich in ring-shaped components as compared to chain systems. To illustrate this, the next system analyzed was toluene–*n*-heptane, where the ring-shaped molecule was substituted for by another in which the symmetry of the ring is broken by the presence of the methyl radical. Figure 2 shows the experimental results on separation obtained in our column at the same working temperatures for the toluene–*n*-heptane system. For comparative purposes, the discontinuous curve represents the plot corresponding to the benzene–*n*-heptane system. As expected, almost the same values are obtained for *n*-heptane-rich mixtures ($x_{\mathrm{Benz}} < 35\%$) for the separation in both systems, and in the converse case of mixtures rich in the other component, considerably higher values are obtained for the benzene–*n*-heptane system than for that involving toluene–*n*-heptane. Such differences point to the great sensitivity of thermal diffusion to small changes in molecular interaction. The position of the maximum on the curve of the toluene–*n*-heptane system is closer to the 0.5 point of the initial volume fraction ($v_0 = 0.57$) and the shape of the curve is more symmetrical than in the case of the benzene–*n*-heptane mixture. All this seems to confirm the foregoing regarding the effect of the greater mobility of ring-shaped molecules. The

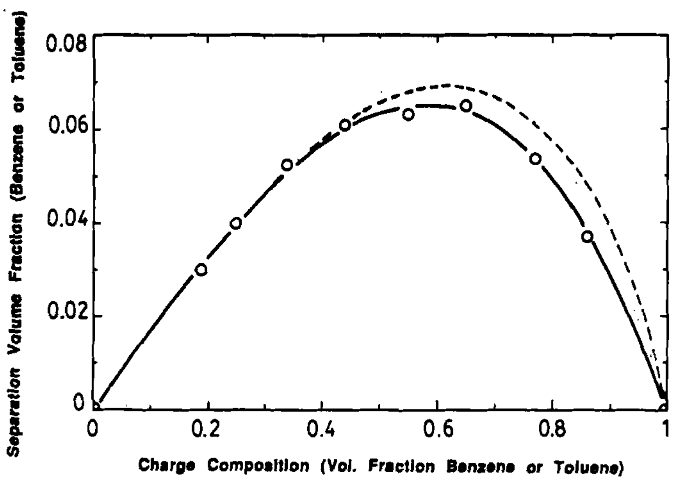

FIG. 2. Separation vs initial composition for toluene–*n*-heptane. The solid line is the best cubic fit to the experimental data. The dashed line represents separation for the benzene–*n*-heptane system.

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toluene molecule has greater "difficulty" than the benzene ring regarding mobility. Thus, the degree of separation between benzene–*n*-heptane is higher than for the toluene–*n*-heptane system, and the shape of the separation/initial concentration curve is less distorted with respect to the equivolumetric mixture.

Finally, Fig. 3 shows the separation results obtained for the carbon

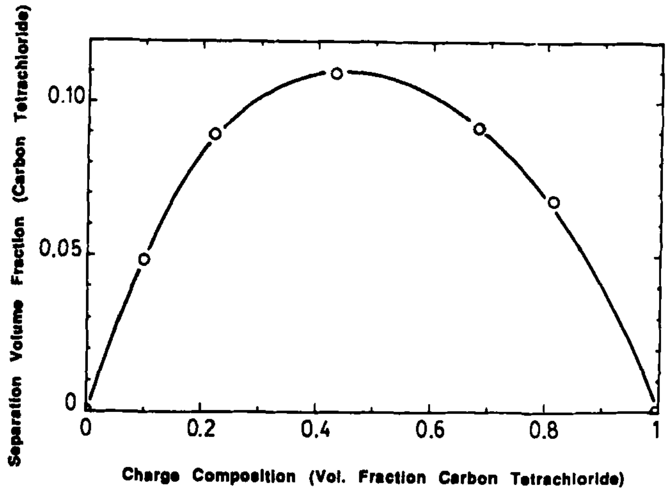

FIG. 3. Separation vs initial composition for carbon tetrachloride–*n*-hexane system. The solid line is the best cubic fit to the experimental data.

tetrachloride–*n*-hexane mixture in our column. It may be seen that for this system, the shape of the curve is similar to a parabola and that the volume fraction corresponding to the maximum is close to 50% ($v_0 = 0.47$). Once again, it is possible to observe the difference in behavior between systems containing benzene as one of the components and the other mixtures. This points out the importance of the different degrees of "mobility" of "rings," "chains," and "tetrahedra" in separation by thermal diffusion and its dependence on feed concentration.

SYMBOLS

L	column length
M	molecular mass
q	separation factor
q_∞	steady-state separation factor
q_∞^0	steady-state separation factor extrapolated to $\Delta T = 0°\mathrm{C}$
r_1	internal radius of external tube
r_2	external radius of internal tube
T_{bp}	boiling point temperature
T_C	cold wall temperature
T_H	hot wall temperature
T_m	mean temperature
ΔT	temperature difference
v	volume fraction
Δv	separation volume fraction
x	molar fraction
δ	solubility parameter
ρ	density

Subscript and Superscript

i	ith component

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Received by editor May 2, 1990
Revision received November 26, 1990

Downloaded At: 12:40 25 January 2011