THE EFFECT OF RANDOM AND SYSTEMATIC ERRORS OF THE EQUILIBRIUM DISTRIBUTION CONCENTRATIONS ON THE NUMBER OF STAGES OF COUNTERCURRENT EXTRACTION WITH VARIOUS CONSUMPTION OF SOLVENT

Ján DojČANSKÝ, Soňa BAFRNCOVÁ and Július SUROVÝ

Department of Chemical Engineering,
Slovak Technical University, 812 37 Bratislava

On using five hypothetical systems differing in the extent of mutual solubility of components, tie-line slope, and type of binodal curve, the effect is evaluated of random and systematic errors in the liquid–liquid equilibrium distribution concentrations on the accuracy of calculated number of theoretical stages of isothermal countercurrent extraction under different operating conditions.

The phase equilibrium data form an indispensable basis of the design calculation of diffusion separation processes. Even if the ways of their obtaining are different, the experimental determination of equilibrium concentrations remains even at present one of the most reliable ones. As every data set obtained experimentally, it will be subject to errors the magnitude of which will influence the results of design calculations in a certain way. For instance, in case of a countercurrent extraction, such a calculation results in the number of theoretical stages, number of transfer units, or efficiency or height of separation equipment.

Starting from the assumption that the equilibrium mole fractions are subjected only to random experimental errors and that the magnitude of these errors is a random quantity with normal probability distribution with a constant value of variance over the whole concentration range, we attempted to obtain some information on the effect of random errors in equilibrium composition on the number of theoretical stages of countercurrent extraction on using the form of simulated measurements of L–L equilibrium composition and their application. The first knowlege of this kind obtained by a study of several hypothetical systems was reported in preceding papers[1,2]. It appeared that the errors in determination of equilibrium distribution concentration of solute, which determined the tie-line slope and position, had the decisive effect on the accuracy of calculated number of theoretical stages and that also the deviation in the calculated number of stages grew with the magnitude of these errors. It was also found out that the inaccuracies in the determination of equilibrium solubility concentrations, by which the ocurse of binodal curve was

described, did not practically influence the number of stages as far as the systems with the closed type of binodal curve were concerned. In case of the systems with open type of binodal curve, their effect was, however, already apparent and the calculated number of thoretical stages was influenced in reduced manner.

Besides the random errors, even the systematic errors play part in the experimental determination of equilibrium data. Their occurrence is especially topical in prediction methods of preparing the equilibrium dependences. The judgement of the effect of systematic errors in the form of a constant relative deviation in distribution coefficients of solute was dealt with in one of our recent papers[3] whereas the systematic errors in the form of an absolute deviation in equilibrium distribution concentrations and their effect on the calculated number of stages in countercurrent extraction was evaluated in the next paper[4]. With the exception of the last work[4], the quantitative description of the effects said was investigated only for certain constant operating conditions of extraction.

In this contribution we will present some new knowledge which makes it possible more complex insight into the problems investigated. It was obtained in the study of effect of random errors in equilibrium distribution mole fractions and of systematic errors in the form of relative error in distribution coefficient of solute on the calculated number of theoretical stages needed for countercurrent extraction under various operating conditions of the process. The operating conditions are represented here by the relative consumption of solvent. The other optional parameters such as the composition of raw material and raffinate product were not changed, remained the same as in preceding papers[2−4], which ensured in all calculations the same concentration range of equilibrium dependences. The form and course of the L–L equilibrium diagrams for the five hypothetical systems is to be seen in Figs $1a-e$.

THEORETICAL

Preparation and Correlation of Equilibrium Data

The types of the model ternary L–L equilibrium systems were chosen to involve a number of differences of systems applied in practice. The etalons of equilibrium data of the depicted model systems with all details were given in preceding paper[2]. They are expressed in terms of values a_{ik} (for $k = 0, \ldots, 3$) by the third-order polynomial

$$\ln K_i = a_{i0} + a_{i1}y_2 + a_{i2}y_2^2 + a_{i3}y_2^3 \tag{1}$$

for component $i = 1, 2, 3$, and where

$$K_i = y_i/x_i \tag{2}$$

is the distribution coefficient. Eq. (1) was later used even for the correlation of experimental equilibrium data. The method of determining coefficients a_{ik} can be found in original works[5,6].

The calculation of composition of equilibrium phases for a chosen value of y_2 is carried out by means of the relations

$$x_2 = y_2/K_2 , \tag{3a}$$

$$x_1 = [1 - K_3 + (K_3 - K_2) x_2]/(K_1 - K_3) , \tag{3b}$$

$$x_3 = [1 - K_2 + (K_2 - K_1) x_1]/(K_3 - K_2) , \tag{3c}$$

and Eq. (2) to determine y_1 and y_3.

The experimental equilibrium data subjected to random errors were prepared by a simulated experiment on the principles of the Monte Carlo method by means of the equations

$$x_{ij,exp} = x_{ij} + \sigma(x_i) N_k , \tag{4a}$$

$$y_{ij,exp} = y_{ij} + \sigma(y_i) N_k . \tag{4b}$$

Eqs (4) are applied for $i = 2, 3; j = 1, 2, ..., l; k = 1, 2, ..., l; x_{ij}$ and y_{ij} are accurate values (etalon) of mole fractions of component i in the phase rich in component
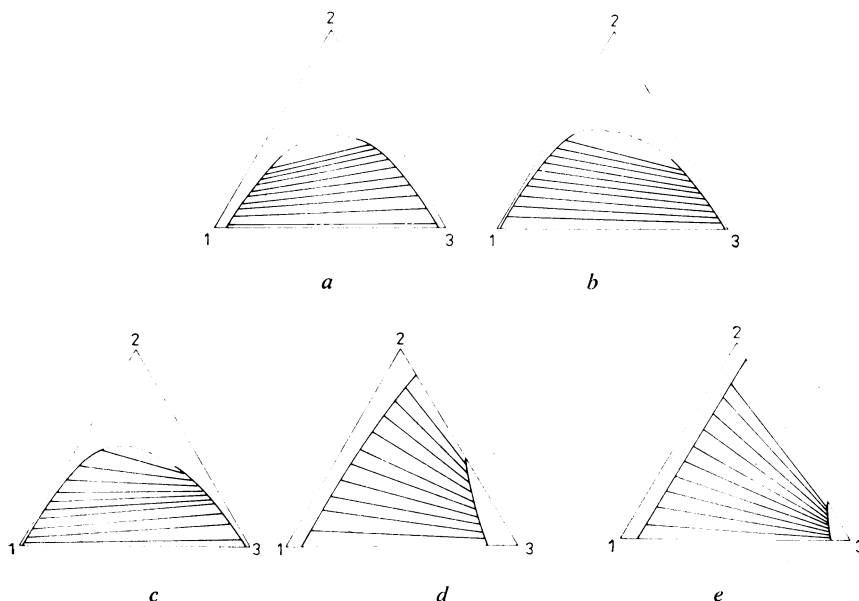

FIG. 1

The L–L equilibrium diagram of hypothetical systems: a I, b II, c III, d IV, e V

1 (x), or in component 3 (y) for which $\sigma(x_i) = \sigma(y_i) = 0$; N_k is a number from the sequence of random numbers with normal distribution, with center $\mu = 0$ and variance $\sigma^2 = 1$; $\sigma(x_i)$, $\sigma(y_i)$ are roots of variance or standard deviations of the mole fraction of component i. Products $\sigma(x_i) N_k$ represent the random error of single measurement. Subscript j is connected with serial number of tie-lines, subscript k with serial number N.

The random errors acquire for the chosen $\sigma(x_i)$ and $\sigma(y_i)$ positive and negative values of different magnitude. Their serial numbers k $(k = 1, ..., l)$ can be still changed as desired before adjoining to the accurate values of mole fractions on keeping the alternation of signs so bringing about the situation allowing to prepare a great number of sets of experimental equilibrium data exhibiting the same values of $\sigma(x_i)$ and $\sigma(y_i)$ but differing mutually in various regroupments of errors. It was shown[2] that such error regroupings manifested themselves in calculating the separation process as differently high deviations in the number of theoretical stages. This knowledge led us to the elaboration of the procedure determining the order of single errors which was applied in this work, too.

When preparing the simulated equilibrium data subjected to systematic errors, a constant value of relative deviation of mole fraction of solute in one of two equilibrium phases or the relative deviation of distribution coefficient following from them was chosen as a measure of their magnitude. The form of binodal curve was retained unchanged in this preparation, i.e., given by the values from the etalon of equilibrium concentrations. From the definition relation of the percent relative deviation for the equilibrium distribution data of mole fractions of solute follows

$$x_{2j,\exp} = x_{2,j}[1 + (d(x_2)/100)] \,, \tag{5a}$$

$$y_{2j,\exp} = y_{2,j}[1 + (d(y_2)/100)] \,. \tag{5b}$$

In Eqs (5) x_{2j} and y_{2j} are accurate values of mole fractions of solute in the phase rich in component 1 (x), or in component 3 (y), respectively; $d(x_2)$ and $d(y_2)$ are the constant values of percent relative deviation of their mole fractions. Mole fractions $x_{3j,\exp}$ and $y_{1j,\exp}$ were determined by linear extrapolation along the binodal curve, whereas fractions $x_{1j,\exp}$ and $y_{3j,\exp}$ by complementing to unity.

Number of Theoretical Stages for Countercurrent Extraction

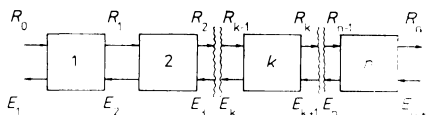The scheme of countercurrent extraction is depicted in Fig. 2. The basis of cal-

FIG. 2

Scheme of countercurrent extraction

culating the number of theoretical stages is the numerical determination of composition of material flows from stage to stage combined with the iteration calculation of composition of equilibrium phases of extract and raffinate. The continuous description of equilibrium is ensured by polynomials (1) whose parameters a_{ik} are identified for every situation by treating the respective set of experimental data. The algorithm of calculating the number of stages was reported in one of preceding papers[6].

Operating Parameters of Process

The solvent consumption is one of optional parameters in solving the counter-current extraction. On its value depends the number of stages needed to reach the required sharpness of separation. The sharpness of separation is given here by the mole fraction of solute in raffinate product $x_{2,n}$ and in the course of calculation, its value remains unchanged. To be able to judge whether and in which way the errors in equilibrium concentrations manifest themselves in calculated number of theoretical stages under changed operating conditions of the process, three values of rela-

TABLE I

The operating conditions of countercurrent extraction and the calculated number of theoretical stages with the etalon of equilibrium data

System	$x_{2,0}$	$x_{2,n}$	$(E_{n+1}/R_0)_{min}$	$\dfrac{E_{n+1}/R_0}{(E_{n+1}/R_0)_{min}}$	N_{TS}
I	0·26	0·01	0·326	1·15	14·38
				1·32	9·18
				1·45	7·53
II	0·50	0·01	0·923	1·15	16·98
				1·30	11·66
				1·45	9·26
III	0·50	0·005	0·730	1·10	5·02
				1·30	3·94
				1·45	3·67
IV	0·50	0·01	1·77	1·15	16·17
				1·24	12·62
				1·45	8·84
V	0·50	0·01	4·96	1·15	23·36
				1·25	16·57
				1·30	14·65

tive consumption of solvent E_{n+1}/R_0 were chosen as a multiple of the minimum value $(E_{n+1}/R_0)_{min}$ for each system judged here. The data on the solvent consumption but also the values of some other optional parameters are given in Table I. In this table, the values of the number of theoretical stages calculated with the etalon of equilibrium data are given, too.

Conditions of Experimental Determination of Equilibrium Data

When preparing the equilibrium data in the form of simulated experiment, it was considered that each set of such data should correspond to ten tie-lines uniformly distributed along the entire heterogeneous region of triangle diagram.

The calculation in terms of Eqs (4) was carried out with constant values of standard deviations of mole fractions of components 2 and 3 chosen from the interval 0.001 to 0.013 considering that $\sigma(x_2) = \sigma(y_2)$ and $\sigma(x_3) = \sigma(y_3)$. Mole fractions of component 1 were considered to be dependent variables and were determined from the conditions $\sum x_{ij,exp} = 1$ and $\sum y_{ij,exp} = 1$ by complementing to unity. The serial numbers of random errors were determined for single systems by the procedure described in detail in preceding work[2]. When preparing the set of equilibrium data for a chosen constant value of $\sigma(x_i) = \sigma(y_i) = 0.003$, the final order of random errors was also found out for which the calculated value of relative deviation in the number of theoretical stages was closest to the mean value of deviations in the number of stages. Fifteen to thirty sets of equilibrium data took part in mean deviation in the number of theoretical stages for single systems. Here it was assumed that the order of errors determined in experiments for $\sigma(x_i) = \sigma(y_i) = 0.003$ would

TABLE II
The values of arithmetic mean $\langle N_{TS,exp} \rangle = \sum N_{TS,exp}/n$, sample standard deviation $s_{NTS} = \sqrt{\sum[(N_{TS,exp} - \langle N_{TS,exp}\rangle)^2/n]}$, mean relative deviation $\langle d_{NTS} \rangle = (100/n) \sum(|N_{TS,exp} - N_{TS}|/N_{TS})$ of the number of theoretical stages in countercurrent extraction and n considered sets of equilibrium data in determining the final order of random errors of equilibrium composition for constant value $\sigma(x_i) = \sigma(y_i) = 0.003$ and relative solvent consumption E_{n+1}/R_0

	System				
Values	I	II	III	IV	V
E_{n+1}/R_0	0.430	1.20	0.803	2.20	5.70
$\langle N_{TS,exp} \rangle$	9.82	12.20	5.16	12.75	21.98
s_{NTS}	0.74	1.3	0.26	2.2	2.4
$\langle d_{NTS} \rangle$	8.5	9.4	4.6	13	9.1
n	27	27	30	26	15

remain the same in the whole range of values $\sigma(x_i)$ and $\sigma(y_i)$ considered here and would not be influenced even by different consumption of solvent in the process. The values of the mentioned characteristic of error distribution are given in Table II.

The calculation in terms of Eqs (5) was carried out with constant values of percent relative deviation of mole fractions of solute chosen from the interval $\pm 2\%$ to $\pm 15\%$. Mole fractions $x_{3j,\text{exp}}$ and $y_{1j,\text{exp}}$ for the considered deviations $d(x_2)$ and $d(y_2)$ are changed only slightly so that they were determined by linear interpolation along the binodal curve.

Each in this way prepared set of equilibrium distribution data was correlated in terms of Eq. (1) before the application itself, by which the continuous description of the equilibrium dependence was obtained .Since in calculating the number of theoretical stages, the experimental equilibrium data are substituted by the correlated data, when evaluating the effect of the magnitude of systematic errors, these are given in the form of mean percent relative deviation of the correlated values of distribution coefficient of solute

$$\langle d(K_2) \rangle = (100/n) \sum_l [(K_{21,\text{corr}} - K_{21})/K_{21}] \qquad (6)$$

for $l = 1, ..., n$ and where $n = 10$. Quantities $\langle d(K_2) \rangle$ have a positive or negative value. The positive value of $\langle d(K_2) \rangle$ is connected with such systematic errors which cause an increase of the $K_{2,\text{corr}}$ values brought about either by an increase of $y_{2j,\text{exp}}$ or a decrease of $x_{2j,\text{exp}}$ in comparison with the accurate values of these quantities. As to the negative value of $\langle d(K_2) \rangle$, the situation is opposite.

RESULTS AND DISCUSSION

The calculated number of theoretical stages based on experimental set of equilibrium data $N_{\text{TS,exp}}$ was compared with the value of N_{TS} obtained with the etalon of equilibrium concentrations in the form of percent relative deviation

$$d_{\text{NTS}} = 100 \, (N_{\text{TS,exp}} - N_{\text{TS}})/N_{\text{TS}} , \qquad (7)$$

by which the basis was obtained for evaluating the effect of random and systematic errors on the accuracy of result of chemical-engineering calculation. The obtained results are presented in Figs 3 and 4 as an graphical illustration of the dependence of deviations in the number of theoretical stages d_{NTS} on standard deviation $\sigma(x_2) = \sigma(y_2)$ which is a measure of magnitude of random errors in equilibrium distribution concentrations. From the nature of random errors of equilibrium data follows that also the deviations in the number of theoretical stages are random quantities. The standard deviations of these quantities from the view of the above-mentioned simplifying assumptions when generating the simulated experimental equilibrium

data acquire values near the values given in Table II. The results of numerical experiment are depicted in Fig. 5 in the form of the dependence of d_{NTS} on mean percent relative deviation of distribution coefficient of solute $\langle d(K_2) \rangle$ which is a measure of magnitude of systematic errors in distribution equilibrium concentrations.

In Figs $3a-c$ it is possible to see the behaviour of systems with closed type of binodal curve towards random errors. Although these systems differ mutually in the slope and orientation of tie-lines (Figs $1a-c$), it appeared[2] that the errors in concentration of component 3 judged according to $\sigma(x_3) = \sigma(y_3)$ from the interval $(0, 0.011)$ did not practically influence the accuracy of calculated number of theoretical stages even in the case when the consumption of solvent E_{n+1}/R_0 was changed. This knowledge made it possible to observe the dependences between d_{NTS} and
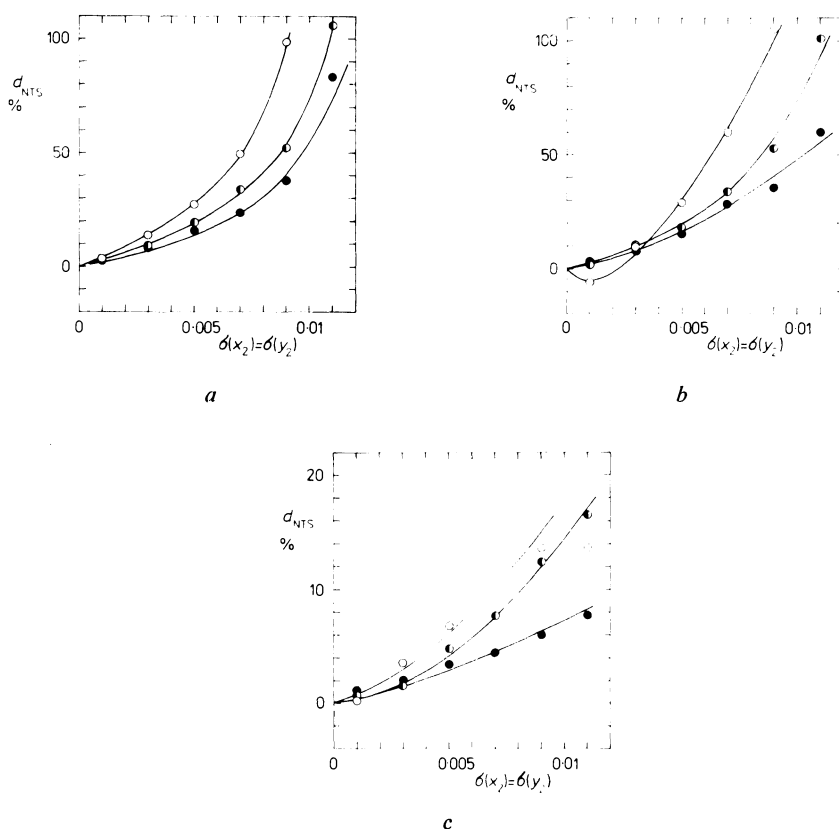

a

b

c

FIG. 3

The dependence of d_{NTS} on $\sigma(x_2) = \sigma(y_2)$ of random errors, ratio $(E_{n+1}/R_0)/(E_{n+1}/R_0)_{min}$: a ○ 1·15, ◑ 1·32, ● 1·45, system I; b ○ 1·15, ◑ 1·30, ● 1·45, system II; c ○ 1·10, ◑ 1·30, ● 1·45, system III

$\sigma(x_2) = \sigma(y_2)$ in Figs 3a − c. Since each of the systems with closed binodal curve was judged at nearly the same multiples of the minimum solvent consumption, their mutual comparison is facilitated.

The behaviour of system I whose tie-lines have positive slope and/or for which $y_2 > x_2$ holds, is given in Fig. 3a. The deviations in the calculated number of theoretical stages increase with increasing errors of equilibrium distribution concentrations, this growth being distinctly weakened with increasing the solvent consumption.

The results of system II whose tie-lines have negative slope and for which $y_2 < x_2$ holds, are illustrated in Fig. 3b. This system behaves similarly to system I providing that the random errors have the value of $\sigma(x_2) = \sigma(y_1)$ greate than 0·003. If, however, the errors of equilibrium data are smaller, the effect of solvent consumption on d_{NTS} is opposite.

Solutropic system III is, in comparison with systems I and II, much less sensitive to the inaccuracies in equilibrium composition, which manifests itself in Fig. 3c in relatively small deviations of number of theoretical stages of countercurrent extraction. It is well-known, and Fig. 1c proves it, that characteristic feature of solutropic systems is that in a certain concentration region it is formed by tie-lines with positive and then with negative slope. And just the occurrence of the tie-lines with negative slope manifests itself in Fig. 3c similarly to system II by a decrease in d_{NTS}. This decrease is, however, much smaller and appears only at low values of $\sigma(x_2)$ and $\sigma(y_2)$.

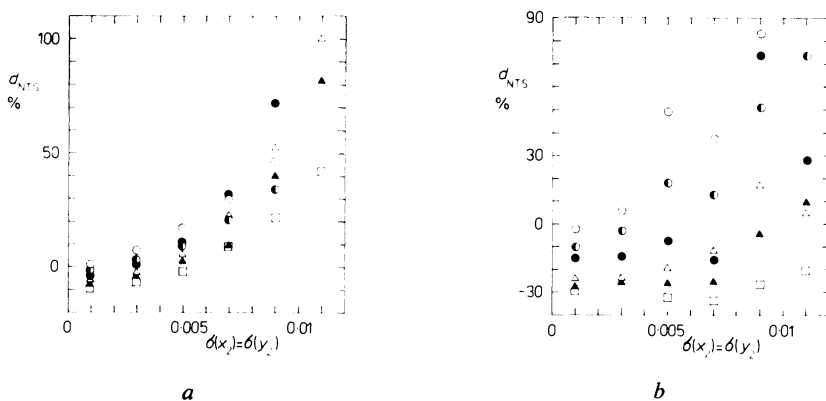The systems with open type of binodal curve, unlike the systems with closed binodal

*a*   *b*

FIG. 4

The dependence of d_{NTS} on $\sigma(x_2) = \sigma(y_2)$ at the following constant values of $\sigma(x_3) = \sigma(y_3)$: ○ 0·001, ◐ 0·003, ● 0·005, △ 0·007, ▲ 0·009, □ 0·011. Ratio $(E_{n+1}/R_0)/(E_{n+1}/R_0)_{min}$: *a* 1·24, system IV; *b* 1·15, system V

curve, behave more complex. It has already followed from the analysis of results of systems IV and V reported in preceding paper[2] that the errors of solubility equilibrium data influence the calculated number of theoretical stages in the opposite direction than that of the effect of errors of equilibrium distribution data. This results in decreasing the number of theoretical stages needed for countercurrent extraction. This decrease is greater with system V, which is probably connected with a lower solubility of components 1 and 2 in 3 manifesting itself in a shorter extract branch of binodal curve. As a result of it, the changes in composition of the extract flows from stage to stage are so small that even smaller values of random errors not only in the distribution but also in the solubility equilibrium data cause great deviations in the number of stages.

Another feature in which differ the systems with open binodal curve from those with closed-loop type is the width of interval in which it is possible to choose the solvent consumption in countercurrent extraction. It was found out that the upper limit of this interval with system IV is about 1·5-multiple of the minimum solvent consumption, with system V is this mutliple about 1·3. The values of the chosen multiplies of $(E_{n+1}/R_0)_{min}$ on examining the given problems are given in Table I. For the sake of clear arrangement, only the results obtained with one value of E_{n+1}/R_0 are illustrated in Figs 4a and 4b. This value is approximately in the middle of the interval mentioned.

A certain aid when judging the effect of random errors of equilibrium distribution concentrations at a constant value of $\sigma(x_3)$ and $\sigma(y_3)$ in Figs 4a and 4b is to notice the change of position of a certain sign in the horizontal direction. In the vertical direction it is possible to examine how the errors of equilibrium solublility concentrations of component 3 at constant values of $\sigma(x_2)$ and $\sigma(y_2)$ influence the deviation in the number of stages. More complex analysis of the behaviour of systems IV and V showed that the increase of solvent consumption brought about a mild increase in the number of theoretical stages and consequently a mild change of values of d_{NTS}. In comparison with the observed changes with systems I and II, they are, however, incomparably lower.

In Figs 5a − e we can see the effect of systematic errors of equilibrium distribution data. The positive values of $\langle d(K_2)\rangle$ are connected with the error causing the increase in K_2 values, which manifests itself, in agreement with expectation, in decreasing the needed number of theoretical stages and consequently in negative values of d_{NTS}. On the left-hand side of the figures, we can see the situation with the opposite orientation of errors. All the five systems behave in a similar way though their equilibrium triangle diagrams are quite the reverse, perhaps only the solutropic system III in Fig. 5c shows an anomaly. In this system, from the view of the lowest of three chosen solvent consumptions, deviations d_{NTS} in the direction of decreasing values of $\langle d(K_2)\rangle$ first increase up to the value of $\langle d(K_2)\rangle$ about -6%, then a strong decrease of d_{NTS} takes place. The observed anomaly takes place apparently as a result
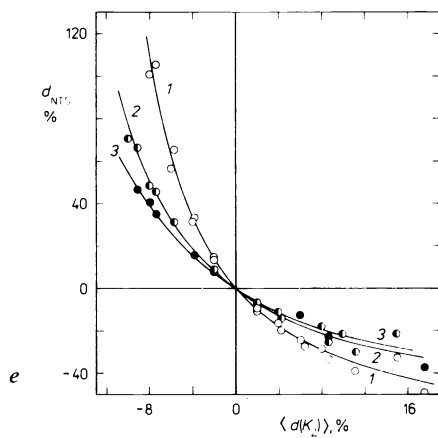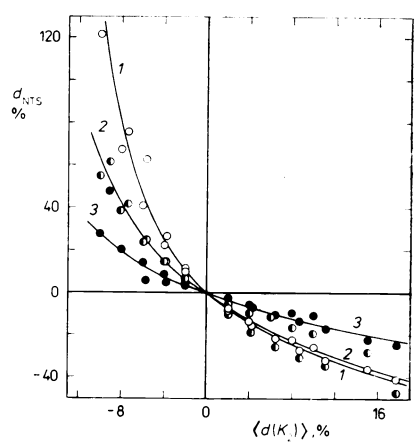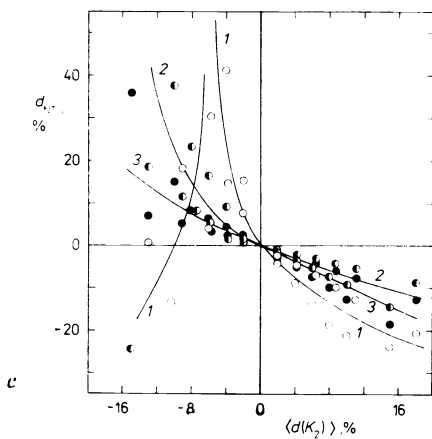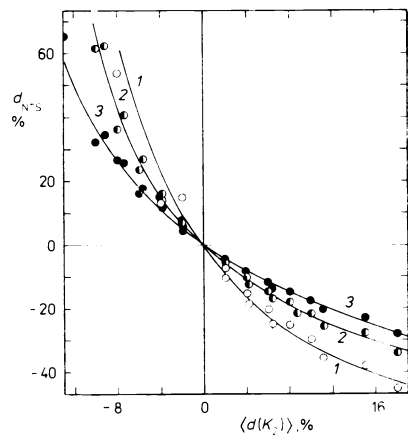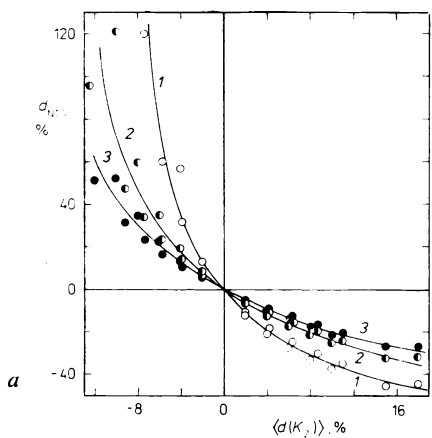
of considerable reorientation of the tie-line arrangement of the system. In case of an increased solvent consumption, however, this anomaly is not observed.

The common feature of the model systems judged here is the fact that the systematic errors causing the decrease of K_2 values influence the inaccuracies in the determination of number of theoretical stages in much higher extent than the errors causing the increase of distribution coefficients K_2. By adapting the operating conditions of countercurrent extraction by increasing the solvent consumption, it is possible to weaken evidently the effect of systematic errors on d_{NTS}.

CONCLUSION

The demands on the accuracy of equilibrium data follow from the importance of result of the calculation itself of the diffusion separation equipment. If we accept, in the design calculation of the number of theoretical stages for countercurrent extraction, the result determined with the accuracy of $\pm 20\%$ as satisfactory, then this requirement can be apparently satisfied when the standard deviation of random errors of equilibrium distribution mole fractions of solute for non-solutropic systems with closed binodal curve does not exceed the values about $0.004 - 0.007$ or if the limiting error in the determination of equilibrium x_2 and y_2 values is not higher than approximately ± 0.8 to 1.4 mole %. The limits of this interval are connected with the amount of solvent used. The effect of errors of equilibrium solubility concentrations is in these calculations insubstantial. In case of the systems with open type of binodal curve, the demands on the accuracy of determination of equilibrium concentrations are higher. They concern especially the systems with small solubility of components in solvent. The value of standard deviation of random errors of not only distribution but also solubility equilibrium mole fractions should not be higher than about 0.003, or the limiting error in the determination of equilibrium composition should not exceed the value of about ± 0.5 mole %.

In case of equilibrium data subjected to systematic errors, one can consider as satisfactory the set of equilibrium data with the magnitude of relative errors in distribution coefficients of solute being in the interval of values -3 to 5% if in the process of countercurrent extraction, the practically lowest acceptable solvent consumption is used. If, however, the used amount of solvent is from the upper limit of the permitted consumption, the demands on the accuracy in determination of K_2 are lower, and the interval of relative errors -6 to 10% is sufficient.

←——

Fig. 5

The dependence of d_{NTS} on $\langle d(K_2) \rangle$ of systematic errors, $(E_{n+1}/R_0)/(E_{n+1}/R_0)_{min}$: *a* 1.15 (*1*), 1.32 (*2*), 1.45 (*3*), system I; *b* 1.15 (*1*), 1.30 (*2*), 1.45 (*3*), system II; *c* 1.10 (*1*), 1.30 (*2*), 1.45 (*3*), system III; *d* 1.15 (*1*), 1.24 (*2*), 1.45 (*3*), system IV; *e* 1.15 (*1*), 1.25 (*2*), 1.30 (*3*), system V

SYMBOLS

a_{ik}	coefficients in Eq. (1)
$d(x_i), d(y_i)$	relative deviation of mole fractions
$d(K_i)$	relative deviation of distribution coefficient
$\langle d(K_i) \rangle$	mean relative deviation
d_{NTS}	relative deviation in number of theoretical stages
$\langle d_{NTS} \rangle$	mean relative deviation
E_k	mole flow rate of extract from k-th stage
E_{n+1}	mole flow rate of extractant
K_i	distribution coefficient
n	number of tie-lines or number of equilibrium data sets
N_k	random number of normal distribution
$N_{TS}, N_{TS,exp}$	number of theoretical stages
$\langle N_{TS} \rangle$	arithmetic mean
R_0, R_k	mole flow rate of raw material or raffinate from k-th stage, respectively
s_{NTS}	sample standard deviation of number of theoretical stages
x_i	mole fraction of component in raffinate phase
$x_{2,0}, x_{2,n}$	mole fraction of extracted component in raw material or in raffinate product, respectively
$x_{ij}, x_{ij,exp}$	accurate or experimental value of equilibrium mole fraction of component in phase rich in component 1
y_i	mole fraction of component in extract phase
$y_{3,n+1}$	mole fraction of component 3 in extractant entering extractor
$y_{ij}, y_{ij,exp}$	accurate or experimental value of equilibrium mole fraction of component in phase rich in component 3
$\sigma(x_i), \sigma(y_i)$	standard deviation of equilibrium mole fractions

Subscripts

corr	correlated
exp	experimental
i	mixture component
j	serial number of experimental point or tie-line
k	serial number of coefficient in Eq. (1) or serial number of random number N or number of arbitrary stage
min	minimum
NTS	referring to number of theoretical stages
TS	referring to theoretical stages
1, 2, 3	system components: 1 initial solvent, 2 solute, 3 solvent

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