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Algorithmic studies of the design of multicomponent, multistage liquid-liquid extraction processes

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Abstract

A design algorithm based on the modified sum-rate algorithm for multi-component, multistage liquid-liquid extraction processes has been proposed. The proposed algorithm was based on existing short-cut design methods and equilibrium-stage model. Comparative study involving the proposed design algorithm and the existing one through their performance algorithms was undertaken with a view to recommending the best algorithm for future use in the determination of the theoretical extraction stages required to achieve a specified degree of separation in a multi-component system. The efficiency of the proposed design algorithm is demonstrated using variety of example problems. In conclusion, the proposed design algorithm has been able to eliminate the deficiencies of the existing short-cut methods regarding the quality of design.

Keywords: design, algorithm, liquid-liquid extraction, modelling, countercurrent

Introduction

Liquid-liquid extraction (LLE) has become one of the more important processes in chemical Engineering (Saien *et al*., 2006) and in several chemical and hydrometallurgical plants. Typical applications of LLE are in metal extraction, aromatics, nitration and sulfunation, polymer processing, waste water treatment, food and petroleum industries (Saien *et al*., 2006). In biotechnological processes, LLE offers the advantage of favourable processing time and easy scale-up for protein recovery and purification (Tambourgi *et al*., 2006). Although LLE has proven to be a reliable and efficient technique (Batterham and Parry, 1996), it is a time, reagent and labour consuming procedure, which cannot be easily automated (Komjarova and Blust, 2006). In fact all these contribute immensely to the capital and operating

cost. In view of this, it is necessary to develop effective and efficient method of design which would exploit the capabilities of modern –day computers to achieve designs that ensure reduction in both fixed capital costs and operating costs while maintaining a friendly interaction with the environment. In the design of liquid-liquid extraction processes, the development of computer methods has hardly risen beyond the level of short-cut methods such as the Smith-Brinkley method (Smith and Brinkley,1960) and Kremser-Sounders-Brown method (Lydersen,1983). Meanwhile, a major difficulty encountered in carrying out designs of LLE lies in obtaining exact solutions to the governing system equations, which are generally large in number and are highly non-linear. A common approach to overcoming this is to employ some simplifying assumptions (model simplifications), thereby avoiding solving the full system equations even though this affects the quality of the design.

Smith and Brinkley (Smith and Brinkley, 1960) developed an analytical short-cut equation, which is applicable to all equilibrium stage processes with any number of stages and handling any number of components. The equation was developed based on the assumption that distribution coefficient (K) and the phase rates are constant throughout the column. Also, the Kremser-Sounders-Brown short-

cut methods (Lydersen, 1983) for design yields approximate solutions of multi-component, multistage extraction design problems (King, 1980). Kremser-Sounders-Brown short-cut method (Lydersen, 1983) was based on the assumptions that the stream flow rates in the column are constant and uses some constant mean value of the distribution coefficients for each component.

In design and performance evaluation, the first step is the choice of specifications. With the specification of independent variables equal to the degree of freedom of the process, the system is determined and the other variables may be found by design computation (Kwauk, 1956). If the number of unknowns equals that of the independent equations in a mathematical model, then the solution is unique and can be obtained.

In this study, the specifications and constraints for the design employed are the number of components (C); the component flow rate for the raffinate feed to stage 1(R_O); the component flow rate for the solvent feed to the stage N (E_{N+1}); the key component and its flow rate in the extract product; the values of temperature of outlet and inlet stream. For performance evaluation, the number of stages (N); the number of components (C); the component flow rates for the raffinate feed to stage 1 (R_O); the component flow rates for the solvent feed to stage N (E_{N+1}); the values of inlet and outlet streams temperature are the specifications and constraints employed. Therefore this paper proposes more efficient methods for the design and performance of LLE processes using existing short-cut methods as a reference point.

Model Formulation

In formulating the models, some assumptions were made for simplification of the governing equations. The LLE process was assumed to be at steady state and adiabatic condition; the aqueous solution to be ideal. The model formulation was based on equilibrium stage model. Fig.1. shows the schematic diagram used in formulating the models.

Equilibrium relationships and material balance

Equilibrium relationships

The equilibrium relationship based on the equilibrium stage model is;

$$Y_{ji} = K_{ji} X_{ji} \; (j = 1,2,...,N \; ; i = 1,2,...,C) \quad \text{----------(1)}$$

where N and C are the number of stages and component respectively; X_{ji} = mole fraction of component i in the raffinate stream leaving stage j; Y_{ji} = mole fraction of component i in the raffinate stream leaving stage j or solvent lean stream leaving stage j and K_{ji} = equilibrium ratio or the distribution coefficient of component i referred to stage j.

By definition,

$$Y_{ji} = E_{ji} / E_j \quad \text{----------(2)}$$

$$X_{ji} = R_{ji} / R_j \quad \text{----------(3)}$$

and

$$K_{ji} = \gamma_{R_{ji}} / \gamma_{ji}^{E} \quad \text{----------(4)}$$

where, E_{ji} ,flow rate of component i in the solvent-rich stream or the extract leaving stage j; R_{ji} ,flow rate of component i in the solvent-lean stream or the raffinate leaving stage j; E_j ,total flow rate of the solvent rich stream or the extract stream leaving stage j; R_j ,total flow rate of the solvent-lean stream or raffinate leaving stage j; γ_{ji}^{R} ,activity coefficient of component i in the solvent-lean stream leaving stage j; γ_{ji}^{E} ,activity coefficient of component i in the solvent-rich stream or extract stream leaning stage j.

Putting (2) and (3) into (1) gives;

$$E_{ji} / E_j = K_{ji} * R_{ji} / R_j \quad \text{----------(5)}$$

and,

$$E_{ji} = K_{ji} * R_{ji} * E_j / R_j \quad \text{----------(6)}$$

where the extraction factor, U_{ji}, is given as

$$U_{ji} = K_{ji} * E_{ji} / R_j \quad \text{----------(7)}$$

And therefore,

$$E_{ji} = U_{ji} R_{ji} \quad \text{----------(8)}$$

Material balance

In line with Fig. 1., the overall material balance for stage 1 to N at steady state is given as:

$$E_1 + R_N = R_O + E_N \quad \text{----------(9)}$$

where, R_O, raffinate feed to stage 1 and E_1, extract stream from stage 1

The component material balance for component i, taken around stage j yields

$$E_{j,i} + R_{j,i} = E_{j+1,i} + R_{j-1,i} \quad \text{----------(10)}$$

The sum equation

Summing the components flow rates for all components for raffinate stream j and extract stream j respectively yields

$$\sum_{i=1}^{C} R_{ji} = R_j \quad \text{----------(11)}$$

and

$$\sum_{i=1}^{C} E_{ji} = E_j \quad \text{----------(12)}$$

Evaluation of K values

According to Perry and Chilton (2007), the distribution coefficient (K) in mole-fraction in liquid-liquid equilibrium ratio is modified as

$$K_{ji} = \frac{Y_{ji}}{X_{ji}} \quad \text{----------(13)}$$

For the fact that this work is based on pre-programmed temperature profile in the stages and for simplicity, the distribution coefficient, K_i was evaluated as a polynomial function of temperature, using the simplified relationship presented by Holland (1975, 1981);

$$\left(\frac{K_i}{T}\right)^{1/3} = a_{1,i} + a_{2,i} * T + a_{3,i} * T^2 + a_{4,i} * T^3$$

----------(14)

where a_1, a_2, a_3 and a_4 are correlation parameters. The values of a_1, a_2, a_3 and a_4 used for the systems tested in this study were generated for each component by solving generated simultaneous equations when K at a given temperature for each component is substituted in equation (14).

Composition update

To compute and update the composition distribution in a stage, the component material balance (equation 10) and the equilibrium relationship (equation 1) were employed. However, for solving the material balance equations, an improved Thomas Algorithm (Wang and Henke, 1966; Boston and Sullivan, 1970) was adopted. Therefore from Fig.1, putting $j = 1,2,...,N$ for the component material balance (equations (8) & (10)) yields

$$\begin{pmatrix} (U_{1,i}+1) & -U_{2,i} & 0 & 0 & \ldots\ldots & 0 \\ -1 & (U_{2,i}+1) & -U_{3,i} & 0 & \ldots\ldots & 0 \\ 0 & -1 & (U_{3,i}+1) & -U_{4,i} & \ldots\ldots & 0 \\ 0 & 0 & 0 & -1 & (U_{N,i}+1) & \end{pmatrix} \begin{pmatrix} R_{1,i} \\ R_{2,i} \\ R_{3,i} \\ R_{N,i} \end{pmatrix} = \begin{pmatrix} Z_1 \\ 0 \\ 0 \\ Z_N \end{pmatrix} \quad \text{----------(15)}$$

Let $Z_1 = R_{0,i}$, $Z_N = E_{N+1,i}$, & $Z_j = 0$, for $j = 2,3,...,N-1$

The algorithm is thus as follows:

1. **Forward Elimination:**

(i) Compute $P_i = U_{1,i}$, $V_i = \dfrac{Z_i}{(U_{1,i}+1)}$ and

$$W_i = \frac{U_{2,i}}{(U_{1,i}+1)}$$

(ii) For ($j = 2,3,...,N-1$)

$$\text{compute } P_j = \frac{U_{j,i} * P_{j-1}}{(1+P_{j-1})}$$

$$W_j = \frac{U_{j+1,i}}{(1+P_j)}$$

$$V_j = \frac{Z_i + V_{j-1}}{(1+P_j)}$$

(iii) Compute $$P_N = \frac{U_{N,i} * P_{N-1}}{(1+P_{N-1})}$$

$$V_N = \frac{Z_N + V_{N-1}}{(1+P_N)}$$

2. **Back Substitution**

(i) Set $R_{N,i} = R_N$

(ii) For ($j = N-1, N-2,...,1$), calculate

$$R_{j,i} = R_j + (W_j * R_{j+1,i})$$

Stream update

To update the extract stream flow rates in this work, equation (12) was employed while updating of the raffinate stream employs

$$R_j = R_O + E_{j+1} - E_1 \quad \text{----------(16)}$$

Temperature update

For temperature update, the sum-rate algorithm proposed by King (1980) which appropriately treats stage temperature as check variables for the enthalpy check function, a modification of the sum-rate which employs a pre-programmed temperature distribution was generated and adopted. Therefore, the temperature update was done using the following procedure:

(i) Specify T_1 and T_N (the temperature for the first and the last stages)

(ii) For a given value of N , compute

$$FACT = \frac{(T_N - T_1)}{N-1}$$

(iii) Compute T_j (for $j = 2,3,...,N$) from

$$T_j = T_{j-1} + FACT \quad \text{----------(17)}$$

The Performance Algorithm

The proposed performance algorithm employed in this study was as a result of modification of the sum-rate algorithm (King, 1980).Therefore the main steps of the modified sum-rates algorithm implemented in this work are given in Fig. 2.

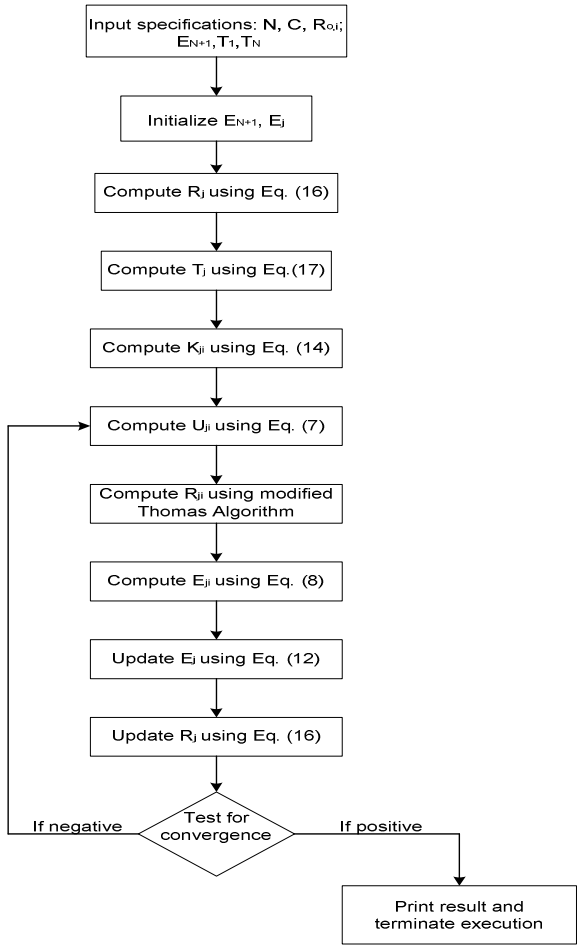

Fig. 2. Flowchart of the performance algorithm implemented

The Proposed Design Algorithm

The proposed design algorithm attempts to solve the mathematical model presented earlier for the Fig.1. without any simplifying assumptions, to determine the value of N which yields a specified value of $Y_{1,i}$ for the key component. The method entails determining the exact key-component mean extraction factor for a given set of stage-by-stage characteristics. This mean extraction factor is then employed on a scheme of the short-cut type to update the configuration

based on which new set of stage characteristics is generated. The procedure is applied iteratively until convergence is achieved for stage configuration as well as stage characteristics. The algorithm as presented here is quite general, although, its application is based on a pre-programmed temperature profile and configuration update method.

The configuration – update method

Re-defining equation (7) with k denoting the key component, which the consideration will be restricted to (that is why the component subscript i is omitted in the relationships) gave

$$U_j = K_{jk} * E_j \Big/ R_j \quad \text{----------(18)}$$

A component balance for stage j enclosing the first stage gave

$$E_{j+1} = R_j + E_1 - R_O \quad \text{----------(19)}$$

And

$$E_{j+1} = \frac{E_j}{U_j} + E_1 - R_O \quad \text{----------(19b)}$$

If $A_j = \frac{1}{U_j}$,

$$E_{j+1} = (A_j * E_j) + E_1 - R_O \quad \text{----------(20)}$$

with j = 1, 2, 3, …, N, equation (20) yields

$$E_2 = (A_1 + 1)E_1 - R_O \quad \text{----------(21)}$$

$$E_3 = (1 + A_1A_2 + A_2)E_1 - (1 + A_2)R_O \quad \text{----------(22)}$$

$$E_4 = (1 + A_1A_2A_3 + A_2A_3 + A_3)E_1 - (1 + A_2A_3 + A_3)R_O$$
$$\text{----------(23)}$$

$$E_{N+1} = \left(1 + \sum_{i=1}^{N}\prod_{j=1}^{N} A_j\right)E_1 - \left(\sum_{i=2}^{N}\prod_{j=1}^{N} A_j + 1\right)R_0$$
$$\text{----------(24)}$$

With

$$\beta = E_{N+1} \quad \text{----------(25)}$$

Equation (24) yields

$$\beta = \left(1 + \sum_{i=1}^{N}\prod_{j=1}^{N} A_j\right)E_1 - \left(\sum_{i=1}^{N}\prod_{j=1}^{N} A_j + 1\right)R_0$$
$$\text{----------(26)}$$

In a design problem, R_O, E_1 and E_{N+1}, are specified, and the goal is to find the value of N, and the corresponding U_j values (deriving from solving the material-balance, equilibrium and summation equations) that satisfy equation(25) . Denoting average U by $\overline{U}$ such that

$$\beta\left(\frac{1}{\bar{U}}\right) = \beta\left(\hat{A}\right) = \beta(A_j) \quad \text{----------(27)}$$

Then,

$$\beta\left(\hat{A}\right) = \beta\left(1 + \hat{A} + \hat{A}^2 + \ldots + \hat{A}^N\right) \quad \text{----------(28)}$$

$$\beta(A) = \left(\frac{A^{N+1} - 1}{A - 1}\right)E_1 - \left(\frac{A^N - 1}{A - 1}\right)R_0 \quad \text{----------(29)}$$

With $f\left(\hat{A}\right) = \beta\left(\hat{A}\right) - \beta$, where β is equal to $\beta(A_j)$ (see equation 27) resulted into

$$f(\hat{A}) = \left(\frac{A^{N+1} - 1}{A - 1}\right)E_1 - \left(\frac{A^N - 1}{A - 1}\right)R_0 - \beta \quad \text{-------(30)}$$

Using Newton's Iteration method to solve equation (30) for Â such that f(Â) = 0 and with Â the most current previous value and for f (Â=1) = 0 gave

$$g(N) = \left(\frac{A^{N+1} - 1}{A - 1}\right)E_1 - \left(\frac{A^N - 1}{A - 1}\right)R_0 - E_{N+1} \text{ -(31)}$$

Solving equation (31) with the condition, g(N) = 0, gave

$$\left(\frac{A^{N+1} - 1}{A - 1}\right)E_1 - \left(\frac{A^{N+1} - A}{A - 1}\right)\frac{R_0}{A} = E_{N+1} \text{----------(32)}$$

Simplification of equation (32) resulted into the value of N given by

$$N = \frac{1}{\log A}\log\left(\frac{(A-1) * E_{N+1} + E_1 - R_O}{(A * E_1 - R_O)}\right) \text{-------(33)}$$

The proposed algorithm

The main steps of the proposed design algorithm are as follows:

(1).Input specifications $E_{N+1,i}$, $R_{O,i}$, T_O, T_{N+1}, key component number (k) and $E_{1,k}$. Set n (iteration counter) to zero and n_c (configuration counter) to zero.

(2).Estimate the starting value of $\bar{U}$ as follows:

$$\bar{E}_i \begin{cases} E_{N+1,i}\,(i=1,2,...,k-1) \\ \frac{1}{2}\left(1+\frac{E_{1k}}{E_{N+1,k}}\right)*E_{N+1,i}\,(i=k,k+1,...,C) \end{cases}$$

$$\bar{R}_i = R_{0i} + E_{N+1,i} - E_i$$

$$\bar{E} = \sum_{i=1}^{N} \bar{E}_i$$

$$\bar{R} = \sum_{i=1}^{C} \bar{R}_i$$

$$\bar{X}_i = \bar{R}_i / \bar{R}$$

$$\bar{Y}_i = \bar{E}_i / \bar{E}$$

$$\bar{T} = \tfrac{1}{2}(T_0 + T_{N+1})$$

$$\bar{K}_i = K_i(\bar{T}, \bar{X}, \bar{Y}) \quad \text{and}$$

$$\bar{U} = K_k \bar{E}_i / \bar{R}$$

Set $N_p = 1$

(3.) Using U from step 2, determine N from equation (33), set

$$R_j = \bar{R}_i, \qquad E_j = E_{jp} = \bar{E}$$

$X_{j,i} = X_i$ and $Y_{j,i} = Y_i$ for all j

(4.) (a) Update T_j

(b) Set $n = n+1$

(5). Compute $K_{j,i} = K_i(T_j, X_j, Y_j)$ and

$$U_{j,i} = \frac{E_j * K_{j,i}}{R_j}$$

(6). Apply the modified Thomas Algorithm to update $R_{j,i}$ and compute $E_{j,i} = U_{j,i} * R_{j,i}$

(7). Update E_j and R_j, compute $X_{j,i}$ and $Y_{j,i}$.

(8). If $N = N_p$, go to step (9), otherwise go to step (11)

(9). If $\frac{1}{N}\sum_{j=1}^{N} E_j - E_{jp} l \le E_{tol}$, *go* to step (10)

Otherwise set $E_{jp} = E_j$ and go to step (4b)

(10). Print results (including n and n_c) and terminate execution.

(11). Set $n = n+1$, compute $U_j = \frac{E_{j,k} * K_j}{R_j}$ and calculate β from equation (29)

(12). Determine $\bar{U}$ from equation (30)

(13). Set $N_p = N$, and update N from Equation (33)

(14). If $N = N_p$, go to step (4b), otherwise, if $N \rangle N_p$, then set $E_{N+1} = E_{N_p+1}, Y_{n+1} = Y_{N_p+1}$, $F_{j+N-N_p} = F_j(j = N_p, N_{p-1},...,2)$,and $F_j = F_i(j = N - N_{p+1}, N - N_p,...,1)$ where F implies (E, R, X, Y) and go to step (4a), otherwise, set $E_{N+1} = E_{N_p+1}$, $Y_{N+1} = Y_{N_p+1}$, and $F_j = F_j(j = 1,2,...,N)$ and go to step (4a).

Results from implementation of the algorithm

Computer software was developed for the proposed algorithms. The software consists of executive programs and subroutines (see Metcalf, 1987). The developed programs were applied to two ternary systems for which general specifications (specifications common to design and performance) are presented in Tables 1.and 2 and specific specifications for the performance and design algorithm are given in Tables 3 and 4. The results obtained are presented in Tables 5-10.

Discussion of Results

Tables 5, 6 and 7 shows that the results obtained from the proposed design algorithm for system 1 are more accurate than the results obtained from the short-cut algorithm. The results obtained from the proposed rigorous design algorithm show a slight deviation from the results obtained from the performance algorithm as shown by their extract stream flow rates in Table 7. This deviation could be as a result of round-off error and truncation error in the execution of the proposed rigorous design algorithm. Tables 8, 9, and 10 give the same deduction as obtained for 5, 6 and 7.The series of deviations being observed in the results could be attributed to poor quality of data used for these systems.

Conclusion

The results obtained showed that the proposed design algorithm is highly efficient and yields more accurate results than the existing short-cut algorithm. Although, the algorithm is very sensitive to the quality of data employed. But it performs better than the short-cut method in terms of quality of design.

Tables and Figures

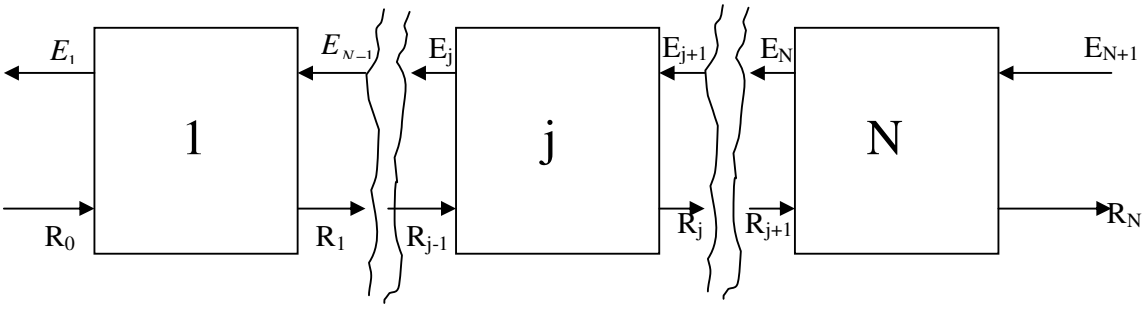

Fig.1: Schematic diagram for counter-current Extraction Column.

Table 1: General specifications for system 1

Component	Extract flow rate entering stage N	Raffinate flow rate entering stage 1
1,1,2 – trichloroethane (solvent)	6.98000	0.01000
Water (solution)	0.83000	5.84000
Acetone (solute)	0.70000	6.34000
Other specification: ETOL=0.001, UTOL=0.001, Temp. of inlet stream=333K, Temperature of outlet stream=330K		

Table 2.: General specifications for system 2

Component	Extract flow rate entering stage N	Raffinate flow rate entering stage 1
Ethyl benzene (solution)	0.83000	0.740000
Styrene(solute)	0.80000	1.340000
Ethylene glycol (solvent)	9.98000	0.01000
Other specification: ETOL=0.001, UTOL=0.001, Temperature of inlet stream=333K, Temperature of outlet stream=330K		

Table 3: Specific specifications for the rating algorithm

System	Number of Stages, N
1	8
2	8

Table 4: Specific specifications for the design algorithm

System	Key Component(k)	Extract flow rate
1	3	0.00000004
2	2	0.00000141

Table 5: Number of stages required for extraction (system 1)

Algorithm	Number of stage, N
Performance algorithm	7
Short-cut design algorithm	10
Proposed design algorithm	8

Table 6: Extract stream flow rates (system 1)

Extract stream flow rate		
Stage	Design	Performance
1	8.53021900	8.50957010
2	8.55110944	8.53021134
3	8.60200112	8.70001004
4	8.70021087	8.73112534
5	8.73102044	8.74201132
6	8.75200234	8.80211111
7	8.80300467	8.90211123
8	8.88280065	----------

Table 7: Extract component flow rates (product stream system 1)

	1	2	3
Performance	7.60718210	0.07896290	0.82342510
Design	7.60814300	0.11882390	0.80325210

Table 8: Number of stages required for extraction (system 2)

	Number of stage, N
Proposed design algorithm	7
Short-cut algorithm	12
Performance algorithm	6

Table 9: Extract stream flow rate (system 2)

	Extract stream flow rate	
Stage	Design	Performance
1	11.59230467	11.6123451
2	11.61304213	11.64304530
3	11.64530274	11.66472116
4	11.66474823	11.67444204
5	11.68157612	11.69241300
6	11.69237463	11.71000000
7	11.72434844	----------

Table 10: Extract component flow rates (product stream system 2)

	1	2	3
Performance	1.15209210	0.12792140	10.33233160
Design	1.14358544	0.19040400	10.25831523

Notation

U_i = Mean Extraction Factor of Component i

A_i = Mean Absorption Factor of Component i

$U_{j,i}$ = Extraction Factor of Component i at Stage j

C = Number of component

i = Component number

j = Stage number

$K_{j,i}$ = Equilibrium ratio of component i at stage j

R_{ji} = Raffinate stream flow rate of component i from stage j (mol/h)

$E_{j,i}$ = Extract stream flow rate of component i from stage j (mol/h)

R_O = Raffinate flow rate into stage 1(mol/h)

E_j = Extract stream flow rate from stage j

E_1 = Extract stream flow rate from the first stage

k = Key component

E_{N+1} = Extract stream flow rate into stage N

$X_{j,i}$ = Mole fraction of component i in the raffinate stream from stage j

$Y_{j,i}$ = Mole fraction of component i in the extract stream from stage j

T = Operating absolute temperature

N = Number of stage

$\gamma_{j,i}$ = Liquid activity coefficient of component i from stage j

π = Multiplication

β = Beta

Σ = Summation

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