Convergent-divergent Column as a Column for the Extraction of Aromatics from Light Petroleum Fraction

T. K. Bandyopadhyay, S. K. Das, and M. N. Biswas*
Chemical Engineering Department, Calcutta University,
92, A. P. C. Road, Kolkata – 700 009, India
*Chemical Engineering Department, Indian Institute of Technology
Kharagpur – 721 302, India

The objective of the investigation is to find the applicability of a convergent-divergent column as an extractor. Experiments have been carried out to extract aromatics from simulated light petroleum fraction in a convergent-divergent column and two straight columns.

Key words:
Extraction; aromatics; Convergent-divergent column; smoke point

Introduction

The increasing demand of the low boiling aromatics for chemicals has provided the primary incentive for the tremendous expenditure by the petroleum industry to improve processing methods. Also, refineries are becoming more aware of applying conventional oil refinery processing methods to the production of petrochemicals. Considerable experience has been gained in the operation of certain types of process units, which are normally utilized to produce these intermediates. The production of aromatic hydrocarbons, i.e., benzene, toluene, and xylene, is increasing every year in the world due to increase in demand for them. The demand for low boiling aromatic hydrocarbons is continually growing. Effluents of catalytic reformers and steam crackers are becoming more and more important as sources of aromatics. Since these mixtures contain other hydrocarbons, which boil in same range of temperatures, and many homogeneous binary azeotropes exist between aliphatics and aromatic hydrocarbons,[1] the recovery of pure aromatics is not possible by conventional distillation. It is therefore necessary to use more elaborate techniques such as solvent extraction or extractive distillation. Generally, in petroleum refineries aromatics are recovered by liquid-liquid extraction using a selective solvent. The aromatics and solvent are then separated by distillation. In most petroleum applications, solvent extraction removes materials of low hydrogen content, principally aromatics from materials of higher hydrogen content. Extraction was first used to upgrade kerosene, which burns with a smoky flame, by removing aromatics. Next, it was used to upgrade lubricating oils by removing aromatic materials with low viscosity indexes. Extraction is also used today to improve charge stocks for catalytic cracking, to improve the quality of light catalytic cycle oils as heating oils, to recover light aromatics from gasoline stocks, to separate i-butene from butane-butene streams, and to remove mercaptans from gasoline. The solvent processes tend to erase the old crude oil marketing system, which only considered a few crude oils satisfactory for lubricant manufacture. By solvent methods, the original properties of the oil can be changed so, that a uniform grade of oil can be manufactured from a wide variety of crude oils.

In literature, the solvents like diethylene glycol,[2–3] triethylene glycol,[4] tetraethylene glycol,[5–6] N-methylpyrrolidone[7–8] and sulfolan,[9–12] etc. are suggested for aromatic extraction.

The different types of extraction columns[13] are used commercially none of them contain convergent-divergent part. The series of convergent-divergent sections of the extractor column have been fabricated for the experimental purpose. This column generates developing flow conditions through the entire length of the contractor. It is well known that the processes of momentum, mass and heat transfer are much intensified under the developing flow condition due to surface turbulence and the slip.[14] The present paper deals with an applicability of a convergent – divergent column to be used as an extraction column for aromatics from simulated light petroleum fraction.

Experimental

The experimental set-up, shown in Fig. 1, consisted of a feed supply line, a mixing chamber, a two-phase pipe line contactor with converging-di-
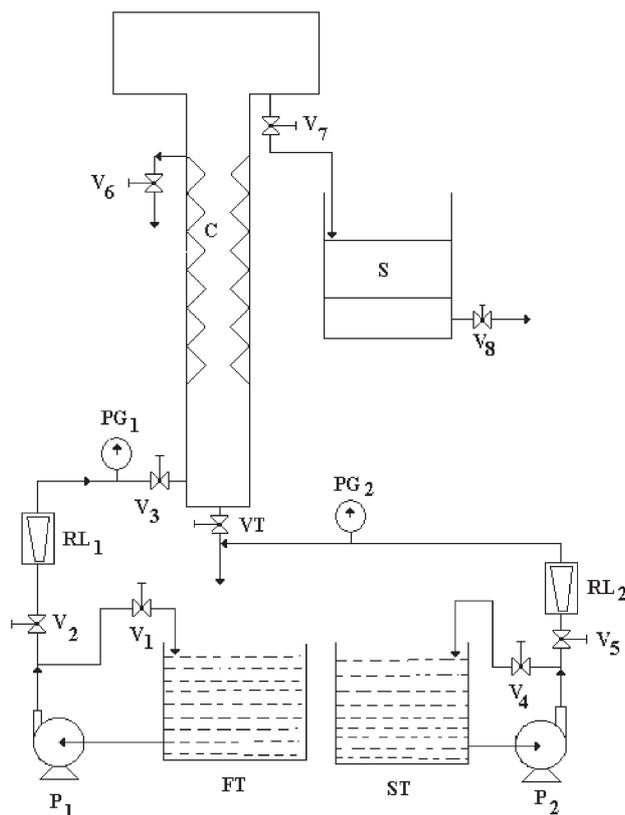
F i g . 1 – *Schematic diagram of the experimental setup*
C – convergent-divergent column; S – separator; FT – feed tank; ST – solvent tank; VT – three way valve, P_1-P_2 – pump; V_1-V_8 – valve; PG_1-PG_2 – pressure gauge; RL_1-RL_2 – rotameter

verging sections, separators and provisions for measuring pressure and collecting samples at different tapping positions along the axis of the pipe line contactor. The feed supply line consisted of a feed tank, feed pump, valves, rotameter, pressure gauge, and a two-way valve. The solvent supply line contained a solvent tank, pump, valves, rotameter, pressure gauge, and a three-way valve. The solvent line was connected to the bottom of the mixing chamber, where as the feed line it was connected to the side of the mixing chamber, and the mixing chamber was connected to the converging-diverging column. The product was taken out from the top of the column through the side valve to the separator. After separation the layers were drawn out separately.

The mixing chamber was fabricated from a Perspex® column of 0.0312 m diameter and 0.0762 m length. The chamber has a bottom opening of 0.011 m for the solvent entry and a side opening of 0.008 m for feed entry.

The test section, i.e., convergent-divergent section of the column was fabricated from Perspex®. The column consisted of 6-converging-diverging sections, each 0.152 m long, forming 6 venturis with the maximum diameter joined together in a series. The minimum diameter of each venturi

(throat-diameter) was 0.0127 m and the maximum diameter was 0.0312 m. The length of each of the convergent and divergent sections was 0.0762 m. The angle between diverging sides was 14°. The same angle was used for converging section. The top of the column had a straight section of diameter 0.0312 m and length of 0.07 m. The sampling port for the withdrawal of the sample for analysis was provided at the end of the converging-diverging section. Experiments were also conducted using a straight tube of 0.0127 m and 0.0312 m internal diameter, and 0.0762 m long two different tubes to compare the performance with the convergent-divergent column.

Feedstock for the investigation of the extraction process was a mixture of kerosene and aromatics. Kerosene was of the superior grade (supplied by Indian Oil Corporation Limited, Haldia Refinery, Haldia, West Bengal, India), and its properties are given in Table 1. The aromatic mixture was prepared by adding analytical grades of benzene, toluene and xylene (supplied by E. Merck (India) Ltd., Mumbai, India). The feed composition is given in the Table 2; the feed contained 35.12 % by volume of the added aromatics. The solvent used for the experiments was diethylene glycol (analytical grade, supplied by E. Merck (India) Ltd., Mumbai, India) and added 2 % (volume) water.

T a b l e 1 – *Properties of kerosene*

specific gravity	0.8014
gravity ^{0} API	45
aromatic fraction, φ %	12.50
smoke point	27 mm

T a b l e 2 – *Composition of the feed*

Constituents	φ %
kerosene	64.88
benzene	19.83
toluene	12.04
xylene	3.25
total aromatic	47.62

Initially, the solvent was pumped to the test column, through the three-way valve in the form of a jet through the nozzle. Then the feed was sent to the test column with help of the feed pump just above the solvent inlet line. The two-phase mixture then passed through the column and was collected in the separator. The raffinate and extract layers

were separated. Next, the raffinate layer was water washed, separated and then the sample was sent for analysis. The water washed raffinate layer was reused for the extraction. The extract layer was sent to the regeneration unit. The regenerated solvent was reused for the extraction. For each run samples were drawn out through the sampling port at the end of the converging-diverging contacting section, separated, and analyzed.

The extraction process was carried out at solvent flow rates and feed flow rates that varied from $0.356 \times 10^{-5} – 24.8 \times 10^{-5}$ m^3 s^{-1} and $2.155 \times 10^{-5} – 17 \times 10^{-3}$ m^3 s^{-1}, respectively.

A batch atmospheric distillation process that was carried out at 200 °C regenerated the extract layer. The regenerated solvent was mixed with the remaining solvent, its properties were determined and the solvent was reused.

The yield of aromatics in raffinate and feed were determined by using fluorescent indicator adsorption technique ASTM D1319–83.[15] Before conducting the experiment, the aromatic content of the feed and the solvent were also measured. The smoke point of the washed raffinate and the feed was measured with the help of standard equipment (ASTM D1322–97[16])

Results and discussion

The aromatic extraction studies have been carried out in two-phase co-current flow, through the converging-diverging section of a vertical contactor for the extraction of aromatics from simulated light petroleum fractions. Similar experiments have also been conducted with two straight columns.

Effect of feed flow rate on extraction of aromatics

Figures 2–4 show the effect of feed flow rate on total aromatics extracted, yield of aromatics extracted, and improvement of the smoke point, respectively. The total aromatics extracted at constant solvent flow rate increases with increase in feed flow rate, which shows that capacity of the system increases with an increase in feed flow rate. The percentage of aromatics extracted at constant solvent flow rate, however, decreases with an increase in feed flow rates. This means that the yield of aromatics extracted decreases with an increase in the capacity of the system. The improvement of the smoke point of the feed decreases with an increase in feed flow rates. So, it shows that increase in feed flow rate increases the capacity of the system but decreases its efficiency.
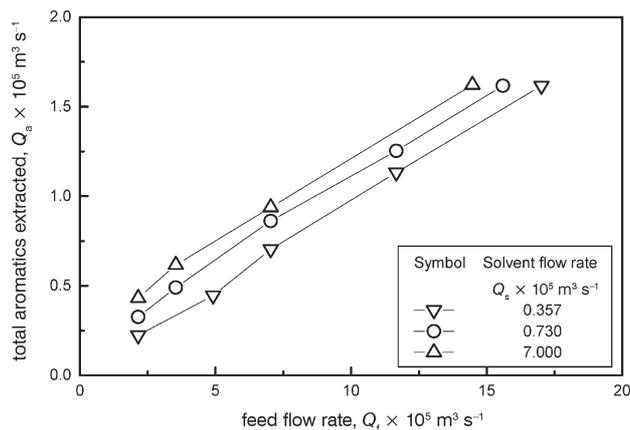

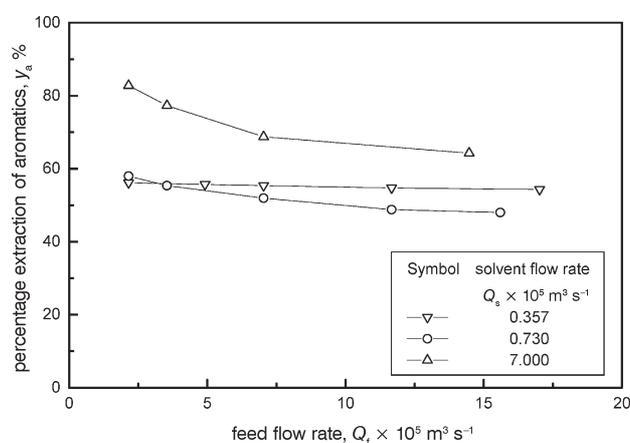F i g . 2 – *Variation of aromatic extraction on the feed flow rate*

F i g . 3 – *Variation of yield extraction of aromatics on the feed flow rate*
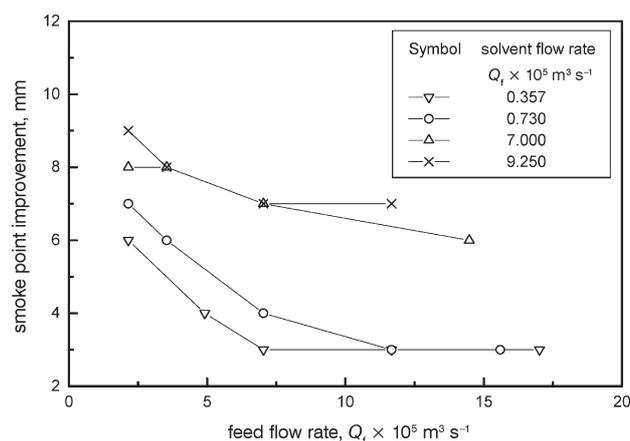

F i g . 4 – *Variation of smoke point improvement on the feed flow rate*

Effect of solvent flow rate on extraction of aromatics

The effect of solvent flow rate on total aromatics extracted, efficiency of aromatics extracted and improvement of the smoke point, is shown in Figures 5–7, respectively. It is evident
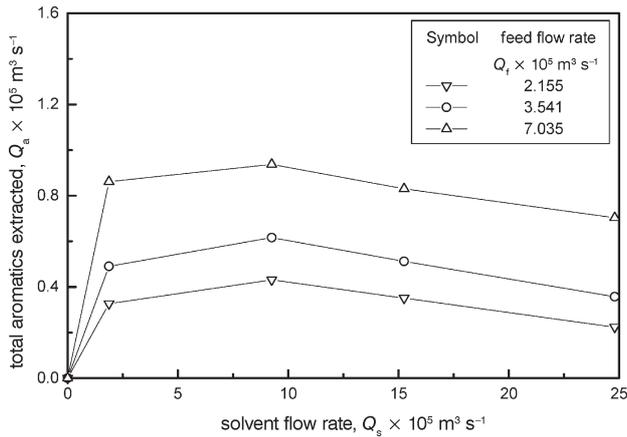
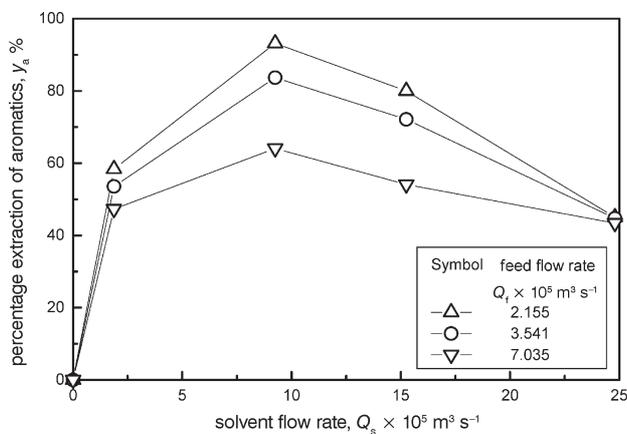F i g . 5 – *Variation of aromatic extraction on the solvent flow rate*

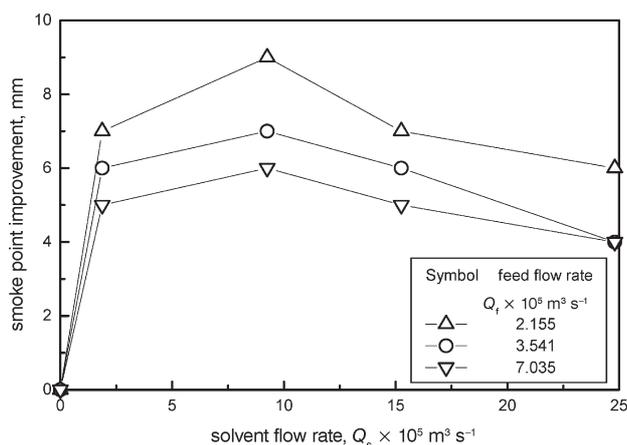F i g . 6 – *Variation of yield extraction of aromatics on the solvent flow rate*

F i g . 7 – *Variation of smoke point improvement on the solvent flow rate*

from these figures that at constant feed flow rate, in all cases there exists an optimum solvent flow rate at which the extraction is maximum. The optimum solvent flow rate is found to be at 9.25×10^{-5} m^3 s^{-1}, independent on the feed flow rate. The maximum percentage of aromatics extracted is 93 %. The maximum smoke point improvement of the product

is 9 mm. All the above values reported are at constant feed flow rate of 2.155×10^{-5} m^3 s^{-1}. This unique phenomenon can be explained by the fact, that the actual residence time of the solvent in the column possibly reduces with an increase in solvent flow rates beyond this optimum value.

Effect of solvent-to-feed flow rate ratio on extraction of aromatics

Figures 8–10 show the effect of solvent-to-feed inlet flow rate ratio on the total aromatics extracted, percentage of aromatics extracted and smoke point improvement, respectively, at constant solvent flow rate. It is seen from Fig. 8 that as the solvent to feed inlet flow rate ratio increases the total aromatics extracted decreases. This decrease is more pronounced at smaller solvent flow rates. Fig. 9 shows that as the solvent to feed inlet flow rate ratio increases the yield of aromatics extracted increases also. This increase is highest at the optimum solvent flow rate. Fig. 10 presents a similar phenomenon. As solvent-to-feed flow rate ratio increases
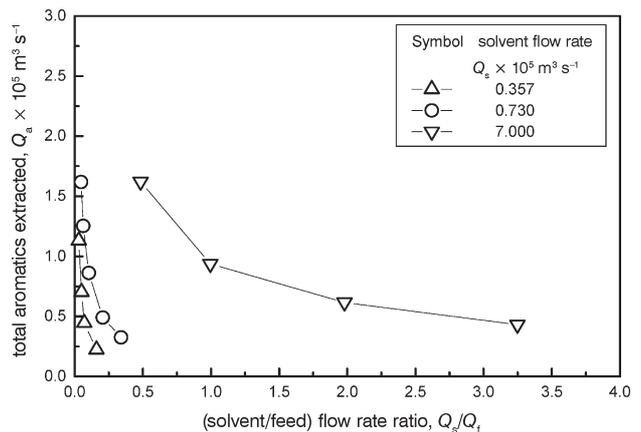

F i g . 8 – *Variation of aromatic extraction on the solvent to feed flow rate ratio*
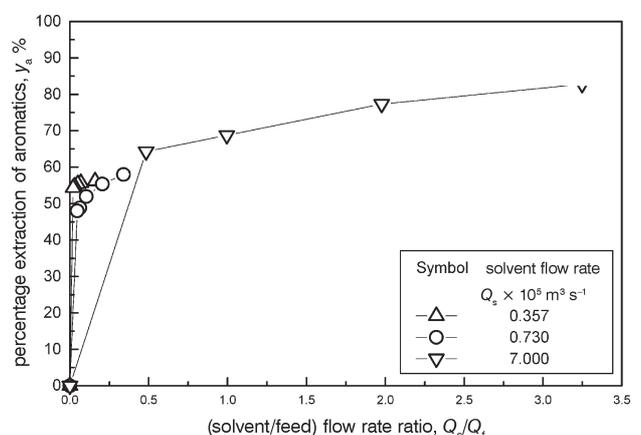

F i g . 9 – *Variation of yield extraction of aromatic on the solvent to feed flow rate ratio*
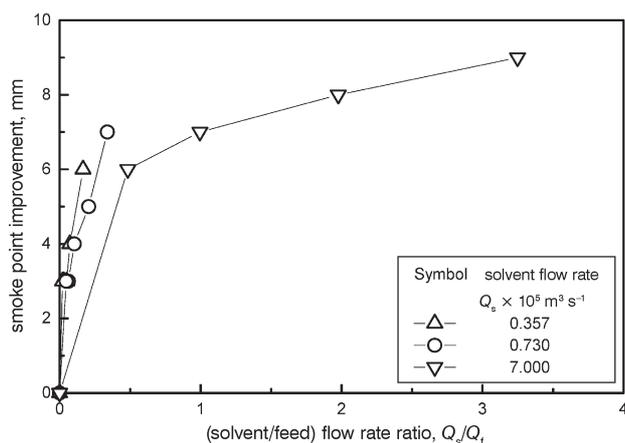
F i g . 1 0 – *Variation of smoke point improvement on the solvent to feed flow rate ratio*

F i g . 1 1 – *Variation of improvement of smoke point with feed flow rate for different extraction column*

smoke point improvement increases, too. The increase in smoke point improvement is more pronounced at lower solvent flow rates.

The capacity of the column increases with the increase in feed flow rate. As the capacity increases, the percentage of aromatics extracted decreases, which is a measure of efficiency of the column. This shows that efficiency is increased at the cost of capacity.

At the solvent flow rate of 9.25×10^{-5} m^3 s^{-1} the percentage of aromatics extracted and the smoke point improvement are both maximum. This behavior can be explained by considering residence time and mixing as parameters. At high solvent flow rate, the residence time is small and efficiency of extraction is small. However, at small flow rate of the solvent the residence time is high but the effect of mixing is small. At high solvent flow rate the extraction is possibly governed by mixing and turbulent mass transfer, whereas at small flow rate only bulk mass transfer controls the extraction so, that the bulk quantity of solvent is the governing parameter (i.e. solvent-to-feed ratio governs the extraction process). However, at intermediate velocity both of these effects govern the mass transfer rate and hence determine the extraction efficiency.

Comparison of the convergent-divergent column with straight columns

Fig. 11 shows the improvement of the smoke point in three different columns. It can be seen from the figure that the performance of the convergent-divergent column is better than that of the straight column. The flow in the convergent-divergent column is developing in nature through the entire length of the contactor, which intensifies the mass transfer in comparison to the straight tubes.
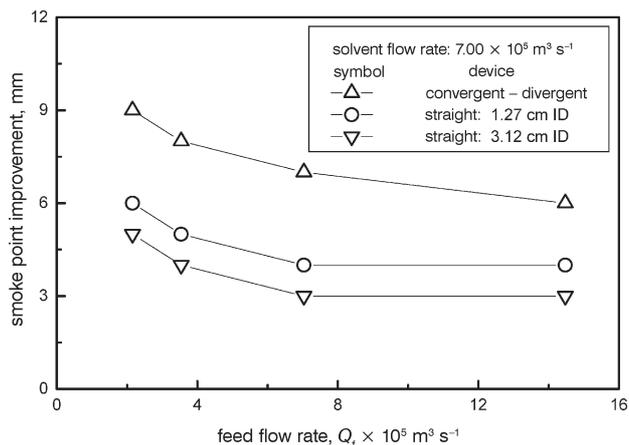
Correlation for convergent-divergent column

The yield of aromatics extracted and the smoke point improvement both depend on the solvent-to-feed flow rate ratio. The curves of yield of aromatics extracted and smoke point improvement initially increase, eventually reach their maximum and then flatten out after certain value of solvent-to-feed inlet flow rate ratio. The maximum yield of aromatics extracted is plotted against corresponding inlet flow rate ratio and shown in Fig. 12. Mathematically this line may be represented as

$$y_{ex} = 70.19 \left(\frac{Q_s}{Q_f} \right)^{0.136}_{\text{critical}} \qquad (1)$$
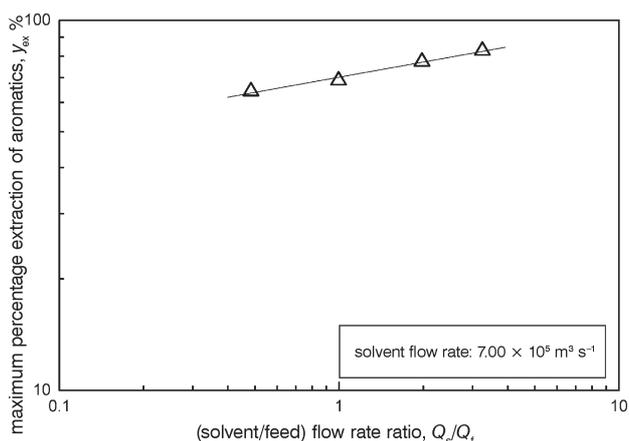

F i g . 1 2 – *Variation of yield extraction of aromatic on the solvent to feed flow rate ratio at optimum solvent flow rate*

Conclusions

Experiments were carried out to test the applicability of the convergent-divergent column as an extractor. The extraction of aromatics from the sim-

ulated light petroleum fraction was carried out using diethylene glycol as solvent. The effects of feed flow rate, solvent flow rate and solvent-to-feed flow rate ratio on the extraction process, were investigated. The extraction of 93 % of aromatics and smoke point improvement of 9 mm were possible in the convergent-divergent column. The comparison of the performance of the convergent-divergent column with straight columns has been done, too, to prove the better efficiency of the former.

List of symbols

Q_f – volume flow rate, $m^3\ s^{-1}$

Q_s – solvent volume flow rate, $m^3\ s^{-1}$

Q_f – feet volume flow rate, $m^3\ s^{-1}$

y_s – yield, Q_s/Q_f, %

y_{ex} – volumen fraction, %

φ – volume flow ratio, Q_{solv}/Q_{feet}

References:

1. *Yorulmaz, Y., Karpuzcu, F.*, Chem. Eng. Res. & Des. **63** (1985) 184.
2. *Read, D.*, Oil Gas J. **51** (1952) 82.
3. *Grote, H. W.*, Chem. Eng. Progr. **54** (1958) 43.
4. *Graham, H. L.*, J. Chem. Eng. Data **7** (1962) 214.
5. *Somekh, G. S., Friedlander, B.I.*, Hydrocarbon Process. **48** (1969)127.
6. *Al-Sahaf, T. A., Kapetanovic, E.*, Fluid Phase Equlibria **118** (1996) 271.
7. *Guccione, E.*, Chem..Eng. **73** (1966) 78.
8. *Nagpal, J. M., Rawat, B. S.*, J. Chem. Technol. Biotechnol. **31**(1981) 146.
9. *Broughton, D. B., Asselin, G. F*, 7th World Pet. Cong., Proc. Sec. **4** (1967) 65, Mexico City, Mexico.
10. *Tripathi, R. P., Ram, A. R., Rao, P. B.*, J. Chem. Eng. Data **20** (1975) 261.
11. *Krishna, R., Goswami, A. N., Nanoti, S. M., Rawat, S. B., Khanna, M. K., Dhoba, J.*, Indian J. Technol. **25** (1987) 602.
12. *Khanna, M. K., Rawat, B. S*, Research and Industry **37** (1992) 67.
13. *http://internet.processmanual.com/ipmcontent/lle_6.htm*
14. *Chadrakar, S. K., Das, S. K., Biswas, M. N.*, Indian J. Technol. **29** (1991) 145.
15. *Standard method of test for Hydrocarbon types in liquid petroleum products by fluorescent indicator adsorption*, ASTM D1319–83, Standard methods for analysis and testing of petroleum and related products 1989, The Institute of Petroleum & John Wiley and Sons, Vol. 1, 156.1–156.8 (1989).
16. *Standard test method for smoke point of kerosene and aviation turbine fuel*, ASTM D1322–97, Standard methods for analysis and testing of petroleum and related products 1989, The Institute of Petroleum & John Wiley and Sons, Vol. 1, 57.1–57.4 (1989).