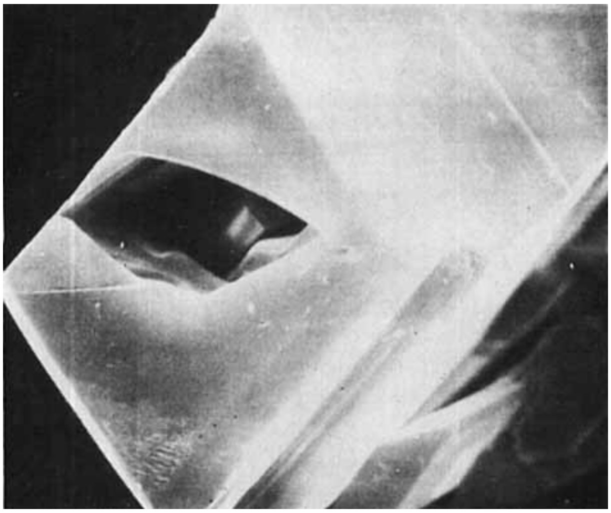

Figure 3. SEM photograph (420X) of aged TPA.

ditions the degree of inclusions in TPA crystals are identical. This suggests that extremely slow growth conditions might be required for the preparation of inclusion-free TPA.

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Calculation Method for Vapor-Liquid Equilibrium in the Critical Region

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The purpose of this work was to test a new prediction method for vapor-liquid equilibrium (VLE) in the critical region. The vapor-liquid equilibrium calculation method used in this study was that due to Leung and Griffiths (1973), which treated composition as a dependent variable. We also followed the work of Moldover and Gallagher (1978), in regard to the power law equations used with the Leung-Griffiths' method.

The input data for the method included the densities of the coexistent phases and the vapor pressure of the pure components along with the critical locus for the binary mixture. Since there are few systems for which the critical locus has been measured thoroughly, the use of the Peng-Robinson (1976) equation of state is investigated for calculation of the critical locus for the mixture in conjunction with this VLE calculational method.

When an equation of state of the van der Waals type is used to calculate VLE near the critical point of binary mixtures, the errors are usually greater than in the other parts of the phase diagram

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(Somait and Kidnay, 1978). While equations may be forced to fit the critical point, the underlying reason for the inability of simple equations of state to accurately predict critical region phase behavior is that these equations assume pressure to be an analytic function of temperature and density (or volume) at and near the critical point, i.e., that pressure is a differentiable function of temperature and volume. The van der Waals type equations of state represent pressure as a third degree polynomial in density whereas, for real pure fluids the relation is of a higher order. Therefore, a more accurate description is accomplished by using power law relations which are found to be accurate in the critical region (Levelt Sengers et al., 1976). These power law relations can be used for binary mixtures by using a corresponding states scheme where the constants of the power laws for mixtures are calculated from the pure component constants.

In classical thermodynamics, VLE calculations are performed by finding the composition of the phases that satisfies the requirements that the fugacities of every component in each phase are equal at the equilibrium pressure and temperature. This method treats composition as an independent variable. The method of Leung and Griffiths is different in that fugacities, pressure, and temperature are treated as independent variables and the densities and compositions of the coexisting phases are dependent quantities. Griffiths and Wheeler (1970), in their formalization of phase equilibria, make a distinction between the various intensive variables which characterize any thermodynamic system. The variables, which take on identical values in equilibrium phases, are called field variables. On the other hand, any intensive variable that has different values across the phase boundary is called a density variable. Examples of field variables are temperature, pressure and chemical potential.

Moldover and Gallagher suggested that the power law relations of pure fluids can be applied to binary mixtures when the critical properties of the mixture are expressed as a function of Leung and Griffith's field variable ζ. This field variable relates the chemical potential of the mixture components at equilibrium and has a value between 0 and 1. The saturated liquid or vapor density is expressed by:

$$\rho = \rho_c\{1 \pm C_1(-t)^{0.355} + C_2 t\} \tag{1}$$

when the (+) sign refers to the liquid and the (−) sign refers to the vapor. The vapor pressure of pure fluids is given by

$$\frac{PT_c}{TP_c} = 1 + C_3(-t)^{1.9} + C_4 T + C_5 t^2 + C_6 t^3 \tag{2}$$

where $t = (T - Tc)/T_c$ and C_1 through C_6 are constants, obtained by fitting the pure fluid properties in the critical region ($0.0001 < |t| < 0.01$). The mixture constants can be calculated by a linear interpolation between the pure component constants.

$$C_i = (C_i^1 - C_i^2)\zeta + C_i^2 \tag{3}$$

where C_i^1 and C_i^2 are the constants of components 1 and 2 respectively. This is essentially a linear mixing rule. Rainwater (1982) observed that this linear interpolation for C_1 gives a larger saturated density difference than that found experimentally. He suggested C_1 should be reduced by the following modification

$$C_1 = \frac{C_1\text{, linear interpolation}}{1 + C_x\zeta^2(1-\zeta)^2\overline{Q}^2(\zeta, t = 0)/\rho_c^2} \tag{4}$$

where C_x is a constant

Equation 4 is based on the assumption that a quadratic combination of reduced density change and mole fraction change across the phase boundary is approximately independent of ζ throughout the mixture. In a subsequent study, Rainwater and Moldover (1983) modified Eq. 4 by changing the quadratic combination to a linear one.

According to Leung and Griffiths, the mole fraction of component 2 in the coexisting phases is given by

$$x = (1-\zeta)\left\{1 - \zeta\left[\frac{\overline{Q}(\zeta,t)}{\rho} - \frac{\overline{Q}(\zeta,t=0)}{\rho_c} - \overline{H}(\zeta,t)\right]\right\} \tag{5}$$

where

$$\begin{aligned}\overline{Q}(\zeta,t) = {} & \frac{P}{RT}\frac{T_c}{P_c}\frac{d(P_c/T_c)}{dx} + \frac{P_c}{RT_c}(C_3'(-t)^{1.9} \\ & + C_4't + C_5't^2 + C_6't^3) + \frac{P_c}{R}\frac{d(1/T_c)}{dx} \\ & \times (1+t)[-1.9C_3(-t)^{0.9} + C_4 + 2C_5't + 3C_6t^2]\end{aligned} \tag{6}$$

where

$$C_1' = C_i^1 - C_i^2$$

The quantity $\overline{H}(\zeta,\tau)$ is found to be identical to zero along the critical locus. An approximate expression for $\overline{H}$ is given by

$$\overline{H} = \frac{C_H}{T_c}\frac{dT_c}{d_x}t \tag{7}$$

where C_H is a constant and can be evaluated from binary vapor-liquid-equilibria data. Thus the intensive state of the system at equilibrium is defined. The adjustable parameters in this method are C_x and C_H.

The method of Moldover and Gallagher requires expressions for the critical locus $(1/T_c, P_c/T_c, \rho_c)$ as a function of composition. The experimental critical property data can be fitted to polynomial expressions and used in the above method. Since there are few systems for which the critical locus has been measured throughly, the Peng-Robinson equation of state (1976) was investigated for calculation of the binary critical locus. While the Peng-Robinson equation of state does not describe the phase behavior in the critical region, it can predict the critical pressure and temperature of mixtures accurately. However, the prediction of the critical densities is less accurate. It was hoped that the Moldover-Gallagher method with the Peng-Robinson critical density locus would give better predictions than the Peng-Robinson equation. The binary interaction coefficient in the mixing rule is obtained by fitting vapor-liquid equilibria data over a wide range of temperature. The method of the critical property calculation is similar to the method used by Peng and Robinson (1977).

The Moldover-Gallagher method with Rainwater's modification (MGR) requires the pure component constants C_1 through C_6, the critical properties and the critical locus as input variables for calculating VLE for binary systems. No attempt was made in this study to adjust the critical locus in order to optimize the fit to the experimental VLE data as was the case in the Moldover and Gallagher work (1978). Their fit to the data was optimized by computer graphics through two adjustable parameters, namely the constant C_H in Eq. 7 which adjusts the value of $\overline{H}$ and the constant C_x which corrects the value of C_1 of Eq. 4.

The MGR method was tested on the following binary systems; propane + n-butane, carbon dioxide + n-butane, methane + carbon dioxide and nitrogen + carbon dioxide. For the first two binary systems, experimental critical locus data were available. For the binary systems, $CO_2 + CH_4$ and $CO_2 + N_2$, the critical locus as calculated from the Peng-Robinson equation of state was used, since no complete data were available. The calculated critical locus was also used with the system CO_2 + n-C_4H_{10} and C_3H_8 + n-C_4H_{10} and the results were compared with VLE data calculated by the experimental critical locus.

PROPANE + n-BUTANE SYSTEM

The experimental data for this system were obtained from Kay (1970). The values of C_H and C_x were 0.0 and 1.0 respectively. As presented in Figure 1, the phase equilibria results of the model were in excellent agreement with experiment, especially in the critical region. It is worthwhile to note the applicability of the method over a wide range of temperature, pressure and vapor-liquid composition using only one set of values for C_H and C_x.

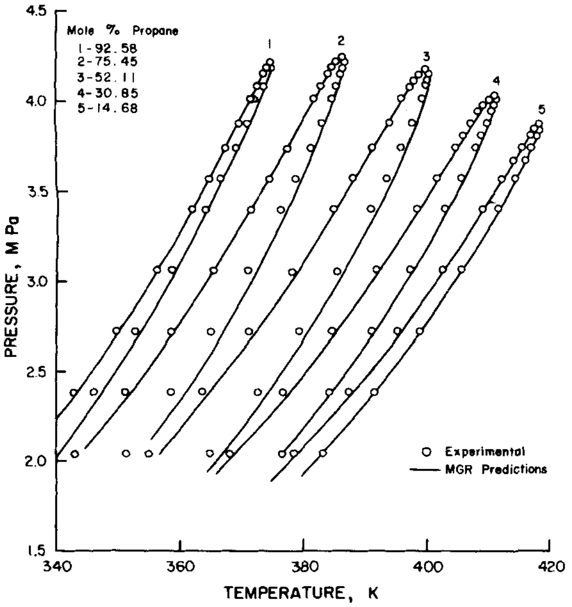

Figure 1. Propane-n-butane system: pressure-temperature diagram.

Vapor-liquid equilibria calculations were done for this binary system using the MGR method with PR critical locus. The binary interaction coefficient used in calculating the critical locus was obtained from the work of Oellrich et al. (1981) as 0.003. The values of C_H and C_x were the same as in the MGR-Experimental locus case. The agreement between experiment and calculation is excellent for all of the dew bubble curves.

The MGR-Experimental locus method yielded an excellent correlation of density, especially in the critical region. The calculated densities by the MGR-PR critical locus method, Figure 2, were greater than experimental values for the liquid and smaller than the exerimental molar densities for the vapor. This result can be understood in the following fashion. The PR critical compressibility factor is 0.307, while the critical compressibility factor for propane is 0.281 and that for butane is 0.274. Thus, PR equations which accurately predict the critical pressure and temperature of propane and butane underestimate the critical densities of these pure components by 8% and 11%, respectively. The MGR method calculates mixture properties by interpolating between the properties of these pure components "parallel to the critical locus." Thus, the MGR method using PR critical densities can be expected to underestimate densities of the coexistent phases of these mixtures by roughly 8–11% of the critical density, in agreement with Figure 2.

CARBON DIOXIDE + n-BUTANE SYSTEM

VLE calculations were performed for this system by the MGR method with the experimental and PR critical loci and compared with the data of Olds et al. (1949). The MGR calculations were also compared with the results of the PR equation of state. The binary interaction coefficient used in the PR critical locus calculation and the VLE data correlation is 0.135. The values of C_H and C_x which gave the best fit for the MGR method are 3.0 and 0.0 respectively. Calculations were performed at 310.93, 344.26, 377.59 and 410.93 K with the last two typical isotherms represented in Figures 3 and 4.

The MGR method with the PR critical loci and experimental critical loci is generally better than the PR equation of state for correlating the VLE data in the critical region of the CO_2 + n-Butane system. The results of the PR equation at lower pressures outside the critical pressure region are excellent.

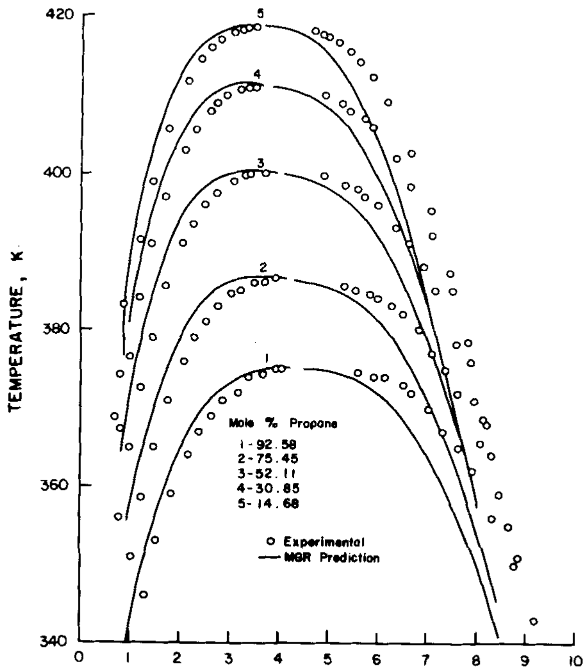

Figure 2. Propane-n-butane system: temperature-density diagram.

METHANE + CARBON DIOXIDE SYSTEM

The MGR method with the PR critical locus method was compared with the correlation of the PR equation. Experimental critical density data were not available for this system. The binary interaction coefficient used in the PR critical locus and VLE data correlation was 0.095. The values of C_H and C_x which gave the best fit for the MGR method were 7.0 and 0.0 respectively except for the 270 K isotherm for which a value of 3.0 was used for C_x. The experimental data were from Al-Sahhaf (1982) and the work of Davalos et al. (1976). The calculations were performed at 219.26, 230, 240, 250 and 270 K. The PR equation of state was shown to be consistently better for this system. The results of the MGR method were inadequate at 219.26 and 230 K isotherms but slightly improved at the 240 and 250 K isotherms. For the 250 K isotherm,

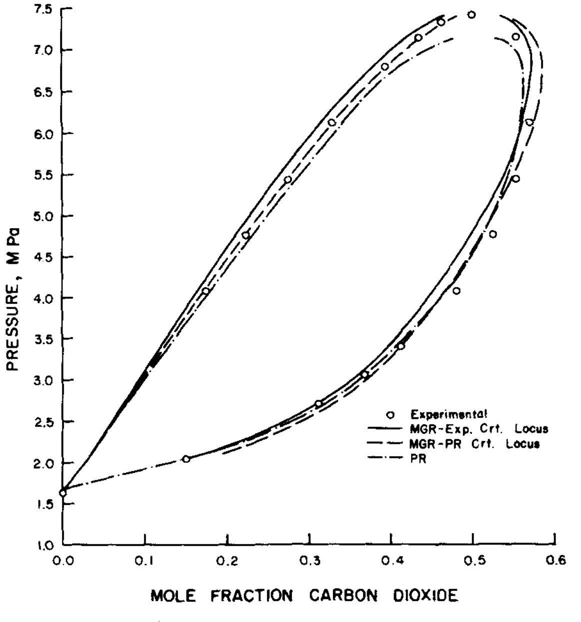

Figure 3. Carbon dioxide-n-butane system: *P-x-y* diagram at 410.93 K.

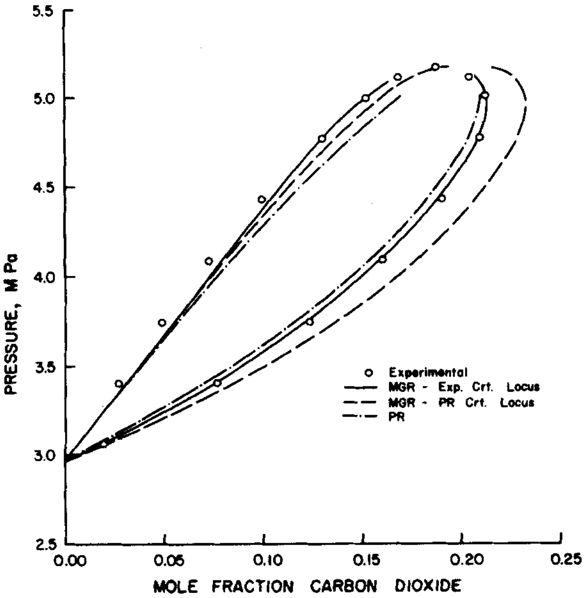

Figure 4. Carbon dioxide-n-butane system: P-x-y diagram at 377.59 K.

the MGR method predicted the liquid phase better than the PR equation. The MGR fit to the data was excellent at 270 K except for the critical region where this method and the PR equation gave *about the* same results.

NITROGEN + CARBON DIOXIDE SYSTEM

Vapor-liquid equilibria calculations were performed for the N_2-CO_2 system at 240 and 270 K. The experimental data were taken from Al-Sahhaf (1982) and Somait and Kidnay (1977). The binary interaction coefficient used for the PR VLE and critical locus calculated had a value of −0.013. The constants C_H and C_x were equal to 0.0 and 2.5 for the 270 isotherm and −7.0 and 0.75 for the 240 isotherm. The PR equation of state gave inadequate predictions in the critical region of both isotherms which was reflected in the erroneous results for the critical locus. Therefore, the MGR method performed inadequately for this system as shown in Figure 5. The PR equation of state gave acceptable results at lower pressures away from the critical region.

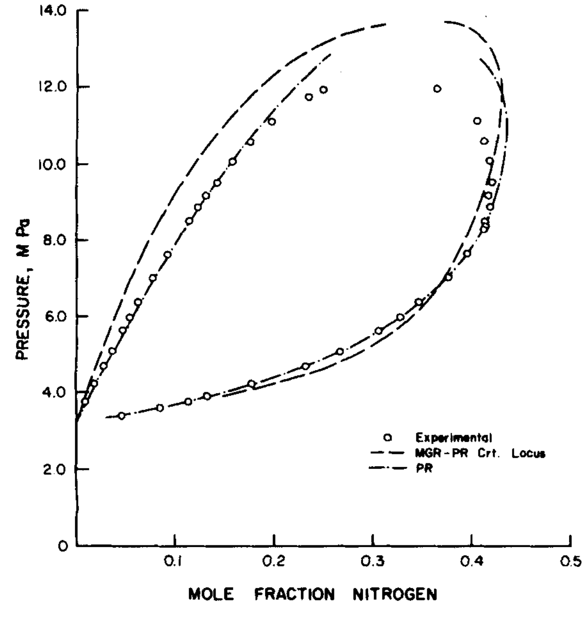

Figure 5. Nitrogen-carbon dioxide system P-x-y diagram at 270 K.

CONCLUSIONS

The MGR method gave a good representation of the critical region for some of the binary systems of this study, but was inadequte for others. The calculation of the isothermal VLE using the MGR method is easier than the case of the Peng-Robinson equation since it does not involve any iterations. The MGR method gave results very close to the critical point where the Peng Robinson equation failed to converge. The MGR method requires accurate representations fo the critical locus. If the MGR methods is used with a critical locus predicted by PR, the densities of the coexistent phases are systematically underestimated. If a better representation of the critical locus were obtained, the overall results would be substantially improved.

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NOTATION

C_1,C_2 = constants in the power law expansion of density
C_3,C_4,C_5,C_6 = constants in the vapor pressure equation
C_H = coefficient in equation for $\overline{H}$
C_x = constant in C_1 Eq. 4
H = function of fugacities on coexistence surface
$\overline{H}$ = function of H and $\overline{Q}$
P = absolute pressure
$\overline{Q}$ = derivative of thermodynamic potential
t = reduced temperatuare $(T - T_c)/T_c$
T = absolute temperature
x = mole fraction in liquid or vapor phase
ζ = fugacity ratio
ρ = density

Subscript

c = critical

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Separation of Isopropyl Ether from Acetone by Extractive Distillation

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Isopropyl ether cannot be completely removed from isopropyl ether–acetone mixtures by distillation because of the presence of the minimum binary azeotrope. Isopropyl ether can be readily removed from mixtures containing this and acetone by using extractive distillation in which the extractive distillation agent is a higher boiling oxygenated, nitrogenous- and/or sulfur-containing organic compound or a mixture of these. Typical examples of effective agents are: dimethyl sulfoxide; sulfolane and propylene glycol; and glycerine, ethylene glycol and adiponitrile.

Extractive distillation is the method of separating close boiling compounds or azeotropes by carrying out the distillation in a multiplate rectification column in the presence of an added liquid or liquid mixture—the liquid(s) having a boiling point higher than the compounds being separated. The extractive agent is introduced near the top of the column and flows downward until it reaches the stillpot or reboiler. Its presence on each plate of the rectification column alters the relative volatility of the close boiling compounds in a direction to make the separation on each plate greater and thus require either fewer plates to effect the same separation or make possible a greater degree of separation with the same number of plates.

When the compounds to be separated normally form an azeotrope, the proper agents will cause them to boil separately during extractive distillation and thus make possible a separation in a rectification column that cannot be done at all when no agent is present. At the bottom of a continuous column, the less volatile components of the close boiling mixtures and the extractive agent are continuously removed from the column. The usual methods of separation of these components are the use of another rectification column, cooling and phase separation, or solvent extraction.

One of the commercially important ways to manufacture acetone is by the catalytic dehydrogenation of isopropanol. Since the acetone does not form an azeotrope with isopropanol and boils 26C° below it, acetone is relatively easy to separate from the unreacted isopropanol by rectification. However, a concurrent reaction takes place in which some of the isopropanol dehydrates to form isopropyl ether. Acetone and isopropyl ether form a minimum azeotrope boiling at 54.2°C and containing 61 wt.% acetone. It is, therefore, impossible to produce pure acetone from acetone–isopropyl ether mixtures by rectification. Extractive distillation would be an attractive method of effecting the separation of acetone from isopropyl ether if agents can be found that will break the acetone–isopropyl ether azeotrope and are easy to recover from the acetone.

The breaking of this azeotrope by extractive distillation is a new concept. The closest application of this concept might be the breaking of the ethanol-water azeotrope. Schneible (1923) used glycerol; Smith (1951) employed ethoxyethanol and butoxyethanol for this purpose; and Catterall (1952) reported gasoline as being effective. These are dehydrations and operate more conventionally as a solvent extraction process. Snyder (1978) has suggested that the selection of the extractive distillation agents be made on the basis of their properties of proton acceptance and dipole moment. To this we would add strong hydrogen bond capability as a major factor. However, even with these guidelines, the search for effective extractive distillation agents must be directed to a large number of compounds and mixtures.

We have ascertained that certain oxygenated, nitrogenous- and/or sulfur-containing compounds, some individually but principally as mixtures, will effectively negate the isopropyl ether–acetone azeotrope and permit the separation of pure isopropyl ether from acetone by rectification when employed as the agent in extractive distillation. Table 1 lists the compounds, mixtures and approximate proportions that we have found to be exceptionally effective. Table 2 lists those that are reasonably successful and Table 3, those that were relatively unsuccessful. The data in Tables 1, 2 and 3 were obtained in a vapor-liquid equilibrium still. In each case, the starting material was the isopropyl ether–acetone azeotrope. The ratios are the parts by weight of extractive agent used per part of isopropyl ether–acetone azeotrope.

The relative volatilities are listed for each of the two ratios employed. For example, in Table 1, one part of ethylene glycol with one part of isopropyl ether–acetone azeotrope gives a relative volatility of 3.80, 6/5 parts of ethylene glycol gives 5.62. One half part of sulfolane mixed with one half part of adiponitrile with one part of isopropyl ether–acetone azeotrope gives a relative volatility of 3.98, 3/5 parts of sulfolane plus 3/5 parts of adiponitrile gives 3.08. One-third part of glycerine plus 1/3 part of DMSO plus 1/3 part of 1,4-butanediol mixed with one part of isopropyl ether–acetone azeotrope gives a relative volatility of 3.46, with 2/5 parts, these three give 3.40.

Several of the compounds and mixtures listed in Tables 1 and 2 and whose relative volatility had been determined in the vapor-liquid equilibrium still were then evaluated in a glass-perforated-plate rectification column possessing 4.5 theoretical plates. The results are listed in Table 4. The data were obtained in the following way. A solution of 460 g of acetone and 40 g of isopropyl ether was placed in the stillpot and heated. When refluxing began, an extractive agent containing pure sulfolane was pumped into the column at a rate of 20 ml/min. The temperature of the extractive