The object of this column is to enhance our readers' collections of interesting and novel problems in chemical engineering. Problems of the type that can be used to motivate the student by presenting a particular principle in class, or in a new light, or that can be assigned as a novel home problem, are requested, as well as those that are more traditional in nature and that elucidate difficult concepts. Manuscripts should not exceed fourteen double-spaced pages and should be accompanied by the originals of any figures or photographs. Please submit them to Professor James O. Wilkes (e-mail: wilkes@umich.edu), Chemical Engineering Department, University of Michigan, Ann Arbor, MI 48109-2136.

# **SOLVENT RECOVERY BY CONDENSATION An Application of Phase Equilibrium and Sensitivity Analysis**

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ondensation is one method that can be used for reondensation is one method that can be used for recovery of a volatile solvent from a gas stream or for similar industrial operations.<sup>[1]</sup> In this method, the temperature (T) of the stream containing solvent vapor (condensable) and an inert gas (noncondensable) is lowered sufficiently to allow (partial) condensation of the solvent vapor. Examples of processes that may use condensation for this purpose are the recovery of tetrachloroethylene  $(C_2Cl_4)$  in dry-cleaning systems, and n-hexane  $(C_6H_1)$  in vegetable oil extraction. Condensation may be part of a more comprehensive system.<sup>[1]</sup>

Apart from heat transfer considerations, the process design conditions for operation of the involved condenser can be guided by application of phase equilibrium and elementary sensitivity analysis.<sup>[2]</sup> Phase equilibrium provides an indication of temperature and composition behaviors during condensation, and the upper and lower limits of variables involved, including the extent of condensation. Sensitivity analysis shows the effects of changing the system operating variables (which are usually within the control of the operator) and any governing constitutive parameter(s); it also provides assessment of the effect of errors in the constitutive parameter(s). $[2]$ 

The purpose of using solvent recovery by condensation from a noncondensable gas as a "class and home problem," apart from illustrating some important aspects of the process, is to demonstrate an actual case for these applications of phase equilibrium and sensitivity analysis. The thermal design of the heat exchanger (condenser), itself, is outside the scope; it is discussed, for example, by McAdams, by Rohsenow, *et*  al., and by Frank.<sup>[3-5]</sup> We first pose a problem statement involving model assumptions and several questions, and then provide solutions to these questions in general terms. Following this, a numerical example is given.

## **PROBLEM STATEMENT**

Figure l shows a schematic flow diagram for partial solvent recovery by condensation. The condenser, C, is depicted as though it were a single-pass, tube-side, horizontal heat exchanger,<sup>[4]</sup> although different configurations involving a vertical arrangement,<sup>[1]</sup> or a dephlegmater,<sup>[5]</sup> can be used. For simplicity, we consider a binary stream consisting of a condensable solvent (component 1,  $e.g., C_2Cl<sub>A</sub>$ ) and a noncondensable inert species (component 2,  $e.g.,$  air or  $N_2$ ). (A condensable species has a critical temperature,  $T_c$ , higher than the stream temperature, and conversely for a



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noncondensable species.)

In Figure 1, the inlet vapor stream (at  $T<sup>in</sup>$ ,  $P<sup>in</sup>$ , and  $x<sup>in</sup>$ , where P is total pressure and  $x_1$  is mole fraction of component 1) enters condenser C, in which  $T<sup>in</sup>$  is lowered eventually to  $T<sup>ex</sup>$ . The resulting two-phase (liquid + vapor) stream enters drum D for separation by gravity into an overhead vapor stream and a bottom stream of liquid 1. Because of frictional pressure drop, the exit pressure from  $D$ ,  $P<sup>ex</sup>$ , is somewhat lower than  $P<sup>in</sup>$ .

The fraction of entering solvent condensed in the exit liquid stream (of C or D) is

$$
f_{1c}^{ex} = \frac{\dot{n}_1^{in} - \dot{n}_1^{ex}}{\dot{n}_1^{in}} = 1 - \frac{\dot{n}_1^{ex}}{\dot{n}_1^{in}}
$$
(1)

where  $\dot{n}_1^{\text{in}}$  is the inlet rate of flow of component 1 (e.g., mol  $s^{-1}$ ) and  $\dot{n}_1^{\text{ex}}$  is the rate of flow of component 1 in the exit vapor stream from D.

#### **ASSUMPTIONS**

We assume the following:

- (A1) The process is at steady-state.
- (A2) The vapor is an ideal-gas mixture.
- (A3) The effect of Pon the molar volume of pure liquid 1 is negligible.
- (A4) The vapor and liquid streams are at thermodynamic equilibrium at any point considered for two-phase coexistence.
- (AS) The solubility of component 2 in liquid I is negligible.
- (A6) The inlet stream to C is single-phase vapor.
- (A7) The coolant in C (water or refrigerant) enters at  $T_{cool}^{in}$ , and there is a nonzero-approach-temperature difference  $(\Delta T_{\text{anor}})$ at the exit of C, where

$$
\Delta T_{\text{appr}} = T^{\text{ex}} - T_{\text{cool}}^{\text{in}} \tag{2}
$$



*Figure 1. Schematic flow diagram for solvent recovery by condensation.* 

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(A8)  $T_{c2} < T^{in}$ ;  $T^{ex} < T_{c1}$ 

(A9) If required, the vapor pressure of component  $1 \, (\, p_1^{\uparrow})$  is represented by the Antoine equation<sup>[6]</sup> in the form

$$
\log_{10} p_1^* = A - \frac{B}{T + C} \tag{3}
$$

where A, B, and C are constants, each  $> 0$ ; values of the constants for  $p_1^*$  in mm Hg and T in °C are given by Dean.<sup>[7]</sup>

#### **.QUESTIONS**

For this problem, we posed the following questions/determinations. The answers are first given in general terms involving chosen system variables and constitutive parameter(s). Numerical values are then used in an example.

- (Q1) For given ( $P<sup>in</sup>$ ,  $T<sup>in</sup>$ ), what is the upper limit of  $x<sub>1</sub><sup>in</sup>$  for the entering stream to be a single-phase vapor?
- (Q2) At what temperature does condensation begin, and does the temperature change as condensation proceeds?
- (Q3) Derive an expression for  $f_{lc}^{ex}$  in terms of a chosen set of system variables, and in so doing establish the number of degrees of freedom (F) among all the quantities chosen for description of the system.
- (Q4) For any condensation to take place in C, what is the range of allowable values (lower and upper limits) of each of the chosen system variables, taken separately?
- (Q5) What are the corresponding lower and upper limits of condensation,  $f_{1c}^{ex}(LL)$  and  $f_{1c}^{ex}(UL)$ , for each of the values in (Q4)?
- (Q6) From the result in (Q3), obtain expressions for all relevant first-order sensitivity coefficients, $[2]$  including for any constitutive parameter(s).
- (Q7) From the results of (Q6), state conclusions about the direction of change of  $f_{lc}^{ex}$  with specified direction of change of each system variable and constitutive parameter.
- (Q8) Express the uncertainty in  $f_{lc}^{ex}$  in terms of the uncertainty of any constitutive parameter.

### **SOLUTIONS**

(Ql) The question stems from assumption (A6). Equilibrium considerations set an upper limit for the inlet composition,  $x_i^{in}$  (UL), in establishing the condition for saturation of the gas stream with respect to component 1. If we equate the chemical potentials of component 1 in vapor and (virtual) liquid phases according to assumption (A4), together with assumptions  $(A2)$  and  $(A3)$ , we obtain

$$
x_1^{in}(UL) = p_1^* (\text{T}^{in})/P^{in}
$$
 (4)

from which  $x_1^{in}(UL)$  can be calculated once T<sup>in</sup> and P<sup>in</sup> are established. Then, typically,  $x_1^{in} < x_1^{in} (UL)$  in actual operation.

(Q2) At the inlet to the condenser, from the discussion sur-

rounding Eq. (4), the partial pressure  $p_1$  of component l is such that

$$
p_1^{in} (= x_1^{in} P^{in}) < p_1^{*}(T^{in})
$$
 (5)

From the inlet to the point at which condensation begins,  $p_1$  remains essentially constant at  $p_1^{in}$ , but  $p_1^*(T)$ decreases as T decreases. Condensation begins at the (initial) dew-point temperature,  $T_{DP}^{init}(x_1^{in},P)$ , where P is the local pressure. This temperature is determined from the condition of equality of  $p_1$  and  $p_1^*$ , analogous to the development of Eq.  $(4)$ 

$$
p_1^* \left[ T_{DP}^{init} \left( x_1^{in} P \right) \right] = p_1 = x_1^{in} P \cong x_1^{in} P^{in}
$$
 (6)

Eq. (6) is a nonlinear relation used to calculate  $T_{DP}^{init}$ from  $x_1^{in}$ ,  $P \cong P^{in}$ , and a vapor pressure relation such as Eq. (3).

As condensation proceeds,  $x_1$  decreases, with consequences for T. At any point between the point of initial condensation and the exit, T may be considered to be a varying dew-point temperature at the local composition and pressure  $T_{DP}(x_1, P)$ . It is determined from a relation analogous to Eq. (6)

$$
p_1^* [T_{\text{DP}}(x_1, P)] = x_1 P \tag{7}
$$

 $T_{\text{DP}}$  can be shown to decrease as x decreases

$$
\frac{\partial \mathcal{T}_{\rm DP}}{\partial x_1} = \frac{\partial \mathcal{T}_{\rm DP}}{\partial p_1^*} \frac{\partial p_1^*}{\partial x_1} = \frac{\partial \mathcal{T}_{\rm DP}}{\partial p_1^*} \left[ \frac{\partial p_1^*}{\partial x_1} + \frac{\partial p_1^*}{\partial P} \frac{\partial P}{\partial x_1} \right]
$$

$$
= \frac{\partial \mathcal{T}_{\rm DP}}{\partial p_1^*} \left[ P + x_1 \frac{\partial P}{\partial x_1} \right] > 0 \quad (8)
$$

Thus, the temperature of condensation of a condensable species from a mixture with a noncondensable species decreases as condensation proceeds. This is in contrast to the condensation of a pure substance (not in a mixture with an inert gas), which occurs at constant temperature.

(Q3) In establishing an expression for  $f_{lc}^{ex}$ , we have a choice of system variables to use, but the number of independent variables—that is, the number of degrees of freedom (F)-is dictated by the difference between the number of these variables (including  $f_{lc}^{ex}$ ) and the number of independent relations among them.

> In Eq. (1), which serves as a material balance for component 1,  $f_{1c}^{ex}$  is an intensive quantity, but it is expressed in terms of extensive flow quantities. To obtain  $f_{1c}^{ex}$  in terms of variables describing the intensive state of the system, we introduce additional variables and relations among them as follows:

$$
\dot{n}_1^{\rm in} = x_1^{\rm in} \dot{n}_t^{\rm in} \tag{9}
$$

$$
\dot{n}_1^{ex} = x_1^{ex} \dot{n}_1^{ex} \tag{10}
$$

where  $x_1^{in}$  and  $x_1^{ex}$  are the mole fractions of compo-

nent 1 in the inlet and exit streams, respectively, and  $\dot{n}^{\text{in}}$  and  $\dot{n}^{\text{ex}}$  are similarly the total molar flow rates

$$
\dot{\mathbf{n}}_t^{\text{in}} = \dot{\mathbf{n}}_1^{\text{in}} + \dot{\mathbf{n}}_2^{\text{in}} \tag{11}
$$

$$
\dot{n}_t^{\text{ex}} = \dot{n}_1^{\text{ex}} + \dot{n}_2^{\text{ex}} \tag{12}
$$

where  $n_2^{in}$  and  $n_2^{ex}$  are the molar flow rates of component 2, as indicated in Figure 1.

From assumption (A5), a material balance on component 2 is

$$
\dot{n}_2^{in} = \dot{n}_2^{ex} \tag{13}
$$

Finally, according to assumption  $(A4)$ ,  $x_1^{ex}$  is constrained by the equilibrium condition analogous to Eq. (4)

$$
x_1^{ex} = p_1^* (T^{ex}) / P^{ex}
$$
 (14)

Eqs. (1) and (9 to 14) are seven equations in 11 system variables [and one constitutive parameter,  $p_1^*(T^{ex})$ ], which seems to suggest  $F = 11 - 7 = 4$ . If, however, *rt:,* an intensive quantity, is independent of flow rate, Fis reduced from 4 to 3. This can be established either by assigning an arbitrary (control or basis) value to, say,  $\dot{n}_1^{\rm in}$ , or by solving Eqs. (1) and (9 to 14) for  $f_{1c}^{\rm ex}$  so as to eliminate all extensive variables and  $x_1^{ex}$  to give

$$
f_{1c}^{ex} = \frac{P^{ex} - p_1^* (T^{ex}) / x_1^{in}}{P^{ex} - p_1^* (T^{ex})}
$$
(15)

Eq. (15) confirms that  $F = 4 - 1 = 3$  and involves the choice of  $x_1^{\text{in}}$ ,  $T^{\text{ex}}$ , and  $P^{\text{ex}}$ , all intensive, as independent system variables.

The extensive state of the system, as given, for example, by the value of  $\dot{n}_1^{\text{in}}$ , involves a scale factor that determines the size of the condenser (not treated here), but does not affect the value of  $f_{lc}^{ex}$ .

(Q4) For condensation to take place,  $f_{lc}^{ex}(LL) < f_{lc}^{ex} < f_{lc}^{ex}(UL)$ , where the lower and upper limits may or may not equal  $0$  and  $1$ , respectively, as might be thought at first. The variables to be investigated are, from  $(Q3)$ , (i)  $x_1^{in}$ , (ii)  $P<sup>ex</sup>$ , and (iii)  $T<sup>ex</sup>$ . We consider the allowable range of values of each of these in tum, for given (allowable) values of the other two.

(i)  $x_1^{in}$ : Setting  $f_{lc}^{ex}(LL) = 0$  in Eq. (15), we obtain

$$
x_1^{in}(LL) = p_1^* (T^{ex}) / P^{ex} (= x_1^{ex})
$$
 (16)

If we set  $f_{1c}^{ex}(UL) = 1$  in Eq. (15), the result can only be satisfied by  $x_1^{in} = 1$ , which is not valid; we conclude that  $x_1^{\text{in}}$  (UL) is constrained by the result of (Q1)

$$
x_1^{\text{in}}(\text{UL}) = p_1^{\ast}(\text{T}^{\text{in}}) / \text{P}^{\text{in}} \tag{17}
$$

(ii)  $P^{ex}$ : Setting  $f_{1c}^{ex}(LL) = 0$  in Eq. (15), we obtain

$$
Pex(LL) = p1* (Tex) / x1in
$$
 (18)

If we set  $f_{1c}^{ex}(UL) = 1$  in Eq. (15), the result can only be

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satisfied by

$$
P^{ex}(UL) \gg p_1^* (T^{ex}) / x_1^{in} \quad \text{or} \quad P^{ex}(UL) \to \infty \tag{19}
$$

(iii)  $T^{ex}$ : The value of  $T^{ex}$  (LL) is set by the thermal condition in Eq. (2)

$$
T^{ex}(LL) = T^{in}_{cool} + \Delta T_{appr}
$$
 (20)

From the result of (Q2), this sets the condition for  $f_{lc}^{ex}$  (UL). To obtain  $T^{ex}$ (UL), we therefore set  $f_{1c}^{ex}(LL) = 0$  in Eq. (15) to give  $T^{ex}(UL)$  implicitly

$$
p_1^* \Big[ T^{ex}(UL) \Big] = x_1^{in} P^{ex} \tag{21}
$$

An explicit expression for  $T^{ex}(UL)$  can be obtained by using a vapor pressure equation, such as Eq. (3), to eliminate  $p_1^*$ .

The above results for (Q4) are collected in Table 1, columns 2 and 3.

(Q5) The results for  $f_{1c}^{ex}(LL)$  and  $f_{1c}^{ex}(UL)$  corresponding to the limits of the system variables are obtained from  $(Q4)$  where noted, and otherwise from Eq.  $(15)$ —in the two cases at  $x_1^{in}$  (LL) and T<sup>ex</sup>(LL). The results are collected in Table 1, columns 4 and 5. The lower limit is O in each case, and the upper limit is less than 1, although it can approach 1 at very high values of pex.

With respect to  $T<sup>ex</sup>$ , the results in columns 2 and 5 emphasize that for the greatest efficiency in solvent recovery, the condenser should be designed so that Tex approaches  $T_{cool}^{in}$ . [5]

(Q6) From Eq. (15),  $f_{1c}^{ex}$  is an explicit function of the system variables  $x_1^{in}$  and  $P<sup>ex</sup>$  and also of the constitutive parameter  $p_1^*(T^{ex})$ . The sensitivity coefficients of  $f_{1c}^{ex}$  with respect to these quantities,  $\partial f_{1c}^{ex}/\partial x_1^{in}$ ,  $\partial f_{1c}^{ex}/\partial P^{ex}$ , and  $\partial f_{lc}^{ex}/\partial p_1^*(T^{ex})$ , can be obtained by direct differentiation. Since  $f_{lc}^{ex}$  is an implicit function of  $T^{ex}$ , through  $p_1^*(T^{ex})$ , the sensitivity coefficient with respect to  $T^{ex}$ is obtained as

$$
\frac{\partial f_{1c}^{ex}}{\partial T^{ex}} \equiv \frac{\partial f_{1c}^{ex}}{\partial p_1^*(T^{ex})} \frac{dp_1^*(T^{ex})}{dT^{ex}}
$$
(22)

The results for all four sensitivity coefficients are listed in Table 2, column 2.

(Q7) One use of the sensitivity coefficients in Table 2 is the information they provide about the direction of change of  $f_{1c}^{ex}$  for a specified direction of change of a system variable or constitutive parameter. This information is contained in Table 2, column 3, which shows the sign of each coefficient, and column 4, which shows the interpretation of the sign. Thus, for the system variables,  $f_{1c}^{ex}$  increases as  $x_1^{in}$  or  $P^{ex}$  increases, and as  $T^{ex}$ decreases; conversely for decreasing  $f_{lc}^{ex}$ .

Achieving favorable values of pex above ambient or the upstream value, by compression, and of  $T<sup>ex</sup>$  below that obtained using cooling water, by refrigeration, is a matter of economic analysis in conjunction with the thermal design of the condenser. It may tum out that even with refrigeration and/or compression, the solvent concentration in the exit vapor stream may not be



low enough to satisfy environmental requirements, or may not be outside explosive limits, if applicable. This may lead to the conclusion that condensation in a particular case is best used as an intermediate stage in a more comprehensive system.<sup>[1]</sup>

(Q8) Another use of sensitivity coefficients is the information provided about uncertainty in a dependent variable, here  $f_{lc}^{ex}$ , caused by uncertainty in a constitutive parameter, here the vapor pressure  $p_1^*$ , which is subject to experimental error. (Values of the independent system variables are set by the investigator and are considered to be without error.) In this case, the uncertainty in  $f_{1c}$ ,  $\delta f_{1c}^{ex}$ , arising from the uncertainty in  $p_1^*$ ,  $\delta p_1^*$ , is given by

$$
\delta f_{1c}^{\text{ex}} = \frac{\partial f_{1c}^{\text{ex}}}{\partial p_1^*} \delta p_1^* \tag{23}
$$

A representative value of  $\delta p_1^*$  is 0.2%.<sup>[9]</sup>

#### **EXAMPLE**

To illustrate use of the results obtained above, consider condensation ofn-hexane (component 1) from an inert gas such as nitrogen (component 2) by means of a water-cooled condenser. The given conditions and calculated results are summarized in Table 3. The vapor pressure,  $p_1^*$ , is calculated from the Antoine equation, Eq. (3), using values of the constants provided by Dean.<sup>[7]</sup> The calculated value of  $f_{1c}^{ex}$  is 0.556 corresponding to  $x_1^{ex}$  = 0.100, which are modest results. [If a refrigerated condenser with T<sup>ex</sup> = -25°C is used,<sup>[5]</sup>  $f_{1c}^{ex}$  is increased to 0.975 and  $x_1^{ex}$ is reduced to 0.006 (results not given in Table 3).] The uncertainty in  $f_{1c}^{ex}$  because of uncertainty in  $p_1^*(T^{ex})$  is negligible, based on Eq. (23), with the value of  $\delta p_1^*$  indicated there and that of

 $\partial f_{1c}^{ex}$  /  $\partial p_1^*$  ( $T^{ex}$ ) from Table 3.

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**TABLE2 First-Order Sensitivity Coefficients (S.C.) of**  $f_{1c}^{ex}$ **:** *Collected Results for (Q6) and (Q7) s.c. (Q6) expression (Q7) sign (Q7)*  for S.C. *of S.C. interp. for*  $f_{1c}$   $\uparrow$  $p_1^*\!\!\left\{\,T\right.^\mathrm{ex}$  $\partial f_{1c}^{ex}$  $+ x_1^{\text{in}} \uparrow$  $\overline{\partial x_i^{in}}$  $(x_1^{\text{in}})^2 [P^{\text{ex}} - p_1^*(T^{\text{ex}})]$  $(1 - x_1^{in})p_1^*(T^{ex})$  $\frac{\partial f_{1c}^{ex}}{\partial P^{ex}}$  $_{\mathbf{p}ex} \uparrow$  $x_1^{\text{in}}\big[\text{P}^{\text{ex}} - \text{p}_1^*\big(\text{T}^{\text{ex}}\big)\big]^2$  $\partial f_{1c}^{ex} = (1-x_1^{in})P^{ex} \left[ dp_1^*(T^{ex})/ dT^{ex} \right]$  $T<sup>ex</sup>$  $\sigma T^{in}$   $\left[\text{e}^{\text{ex}}-\text{p}_1^*(\text{T}^{\text{ex}})\right]^2$  $\frac{\left(1-x_1^{\text{in}}\right)P^{\text{ex}}}{x_1^{\text{in}}\left[P^{\text{ex}}-p_1^*\left(T^{\text{ex}}\right)\right]^2}$  $\partial f_{1c}^{ex}$  $p_1^*(T^{ex})\downarrow$ 

## **TABLE3 Example: Condensation of n-hexane (1) from Inert Gas**  *Given Conditions Calculated Results*   $x_1^{in} = 0.200$  $f_{1c}^{ex} = 0.556$  $T^{in} = 50^{\circ}C$   $x_1^{ex} = 0.100 = x_1^{in}(LL)$  $P^{1n} = 2.5 \text{ bar}$   $x_1^{1n}$  (UL)=0.216  $f_{1c}^{ex}$  (UL)= 0.597 at  $x_1^{in}$  (UL)  $P^{ex} = 2.2 \text{ bar}$  $T_{cool}^{in} = 22^{\circ}C$  $T_{\rm DP}^{\rm init}$  = 47.8°C  $\Delta T_{\text{appr}} = 5K$  $P^{ex}(LL)=1.1$  bar (at given  $x_1^{in},T^{ex}$ )  $p_1^*({\rm T}^{\rm in})=0.541{\rm bar}^{\dagger}$  $T^{ex}(UL)=44.4^{\circ}C(at\, given\, x_1^{in},P^{ex})$  $\partial f_{1c}^{ex}/\partial x_1^{in} = 2.78$  (at given conditions)  $p_1^* (T^{ex} = 27^\circ C) = 0.220 \text{ bar}^{\dagger}$  $\partial f_{1c}^{ex}$  /  $\partial P^{ex}$  = 0.224 bar<sup>-1</sup>  $\partial f_{1e}^{ex} / \partial T^{ex} = -0.0213 K^{-1}$  $\frac{\partial f_{1c}^{ex}}{\partial p_1^*}$  /  $\frac{\partial p_1^*}{\partial p_2^*}$  = -2.245 bar<sup>-1</sup>  $\dagger$  In Eq.(3) for  $p_1^*$ , the Antoine constants are  $^{[7, p. 5.48]}$ : A=6.87601; B= 1171.17; C=224.41

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