

AIChE MI

MODULAR INSTRUCTION

Series B:

STAGEWISE AND MASS TRANSFER OPERATIONS

Volume 6:
Separation
Processes



AMERICAN INSTITUTE OF CHEMICAL ENGINEERS

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**STAGEWISE AND
MASS TRANSFER
OPERATIONS**

Volume 6:
Separation Processes

J. M. Calo and E. J. Henley, Series Editors



AMERICAN INSTITUTE OF CHEMICAL ENGINEERS

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INTRODUCTION

In 1975 a new venture in education by and for the chemical engineering community was initiated. Prepared by the CACHE Corporation (Computer Aids for Chemical Engineering Education) and under the sponsorship of the National Science Foundation (Grant HES 75-03911), a series of small self-study fundamental concept modules for various areas of chemical engineering were commissioned, Chemical Engineering Modular Instruction, CHEMI.

It has been found in recent studies that modular study is more effective than traditional instruction in both university and continuing education settings. This is due in large measure to the discrete focus of each module, which allows the student to tailor the speed and order of his or her study. In addition, since the modules have different authors, each writing in his or her area of special expertise, they can be produced more quickly, and students may be assured of timely information. Finally, these modules have been tested in the classroom prior to their publication.

The educational effect of modular study is to reduce, in general, the number of hours required to teach a given subject; it is expected that the decreased time and expense involved in engineering education, when aided by modular instruction, will attract a larger number of students to engineering, including those who have not traditionally chosen engineering. For the practicing engineer, the modules are intended to enhance or broaden the skills he or she has already acquired, and to make available new fields of expertise.

The modules were designed with a variety of applications in mind. They may be pursued in a number of contexts: as outside study, special projects, entire university courses (credit or non-credit), review courses, or correspondence courses; and they may be studied in a variety of modes: as supplements to course work, as independent study, in continuing education programs, and in the traditional student/teacher mode.

A module was defined as a self-contained set of learning materials that covers one or more topics. It should be sufficiently detailed that an outside evaluation could identify its educational objectives and determine a student's achievement of these objectives. A module should have the educational equivalent of a one to three hour lecture.

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Volume 6. Separation Processes

- B6.1 Degrees of Freedom by Description Rule
- B6.2 Degrees of Freedom by the Design Variable Method
- B6.3 Selection of Separation Processes
- B6.4 Ideal Stages and Stage Arrangements
- B6.5 Ideal Cascade Calculations
- B6.6 Introduction to Crystallization
- B6.7 Continuous Crystallization
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CONTENTS

	INTRODUCTION.....	iv
B6.1	DEGREES OF FREEDOM BY DESCRIPTION RULE..... L. R. Hile	1
B6.2	DEGREES OF FREEDOM BY THE DESIGN VARIABLE METHOD Joseph M. Calo	7
B6.3	SELECTION OF SEPARATION PROCESSES Ho-Lun Lee	17
B6.4	IDEAL STAGES AND STAGE ARRANGEMENTS R. A. Mischke	24
B6.5	IDEAL CASCADE CALCULATIONS J. L. Borowitz and D. Wolf	30
B6.6	INTRODUCTION TO CRYSTALLIZATION P. M. Schierholz and S. M. Gwynn	39
B6.7	CONTINUOUS CRYSTALLIZATION P. M. Schierholz and S. M. Gwynn	47
B6.8	DIALYSIS..... V. L. Vilker	53
B6.9	REVERSE OSMOSIS AND ULTRAFILTRATION V. L. Vilker	62
B6.10	FLOTATION..... Robert K. Prud'homme	70
B6.11	CYCLIC SEPARATIONS—PARAMETRIC PUMPING, PRESSURE SWING ADSORPTION AND CYCLING ZONE ADSORPTION Phillip C. Wankat	87
B6.12	ZONE MELTING—FRACTIONAL SOLIDIFICATION William R. Wilcox	101
	APPENDIX: SOLUTIONS TO THE STUDY PROBLEMS.....	107

Solutions to the Homework Problems are available as a separate reprint from the AIChE Educational Services Dept., 345 East 46th St., New York, NY 10017. The cost is \$5.00.

Degrees of Freedom by Description Rule

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OBJECTIVES

At the completion of this module, the student should be able to:

1. Determine the number of variables needed to properly define multistage separation operations.
2. Test separation problem statements for improper definition and and modify them accordingly.
3. Construct problem definitions appropriate to practical goals, such as designing or operating a distillation column.
4. Distinguish dependent from independent variables in formulating a separation problem.
5. Explain the purpose of the elements used in separation operations, such as valves, heat exchangers, pumps, and stages.

6. Construct and interpret flow chart sketches of separation operations.

PREREQUISITE MATHEMATICAL SKILLS

1. None

PREREQUISITE ENGINEERING AND SCIENCE SKILLS

Familiarity with:

1. General types of separation operations and process equipment (absorbers, distillation columns, reboilers, heat exchangers, etc.).
2. Terminology related to separation calculations (minimum stages, reflux, equilibrium stage, saturated, subcooled, relative volatility, etc.).
3. How pertinent variables influence separation operations (composition, flow rate, pressure, temperature, etc.).

INTRODUCTION

How much information is enough to solve a problem? Students generally don't give this question much thought, since teachers always seem to provide just enough data to solve homework problems. However, in the industrial world, data sheets are likely to be blank! That is, engineers may be required to identify the pertinent data needed to solve the problem. In a converse situation, management may have a whole list of desired specifications for a process the engineer is to design. The question then becomes: Is *too much* data supplied?

This module will explain how to determine the correct number of variables ("degrees of freedom") which uniquely define a multistage process problem. Students will also gain practice in selecting which variables may be specified for some typical problems, such as design of a new distillation column or operation of an existing unit. Additionally the method used (description rule) gives a feeling for the physical, real-world side of separation processes such as absorption, stripping, distillation, extraction, etc.

WHAT ARE "DEGREES OF FREEDOM?"

The problem: Solve for unknowns x and y , given the information $x + y = 10$. Clearly there is not enough in-

formation to solve this uniquely. The problem is "under-specified." There are an infinite number of possible solutions. If one more piece of information was supplied about x and/or y , then the problem would be "uniquely specified." The number of additional pieces of information needed to uniquely specify a problem is termed by various authors as the "design variables" or "degrees of freedom" of the problem. In general, the following holds:

$$\left[\begin{array}{c} \text{number} \\ \text{degrees of} \\ \text{freedom} \end{array} \right] = \left[\begin{array}{c} \text{number of} \\ \text{unknowns} \\ \text{in problem} \\ \text{as stated} \end{array} \right] - \left[\begin{array}{c} \text{number of} \\ \text{independent} \\ \text{equations} \\ \text{relating} \\ \text{unknowns} \end{array} \right] \quad (1)$$

If there are more unknowns than equations relating them, there would be only two ways to solve the problem—find some more equations or arbitrarily assign values to some

of the unknowns. The former choice is rarely possible in real problems, since the "equations" are expressions of physical laws, such as material balances, etc. Thus, engineers are left with some "freedom" to choose variables, which is really what "design" is all about—hence the terminology.

Let's look at another, but related situation. Find x and y given:

$$x + y = 10$$

$$x - y = 6$$

$$x - 3y = 4$$

It is generally not possible to solve a problem with more equations than unknowns (if all equations are truly *independent*). Such a problem is termed "over-specified."

Now, one might say: "All that's perfectly obvious. I'd never waste my time trying to solve such underspecified or overspecified problems." Yet, problems in separation processes become much more complicated than these simple examples. It becomes extremely useful to have a tool which guarantees that there *is* a solution to the problem being worked on. Whether or not one can *find* that solution is another question!

COUNTING DIRECTLY CONTROLLABLE VARIABLES—THE DESCRIPTION RULE

Clearly it would be extremely tedious if every time an engineer was faced with a separation problem, he or she had to count up all the equations (material balances, heat balances, composition restrictions, relations of enthalpy to temperature, equilibrium relations, etc.) and all the unknowns (compositions, flow rates, pressures, temperatures, heat flows, numbers of stages, etc.) and take the difference, as in Equation 1, to find the degrees of freedom needing specification, in order to define the problem. Alternate methods have been developed which are simpler. One of these is the Kwauk or design variable method, described in the next module. One very different approach, which is lesser known, but has advantages in that it is simple, quick and develops physical insight, is one formulated by D. N. Hanson (1). Hanson shifts the viewpoint from the abstraction of equations and unknowns to the actual physically operating separation process (or at least our mental picture of it). For example, imagine operating a distillation column. To achieve a desired separation, there are certain things one can *control* by operation or construction. Appropriate valves can be turned adjusting flow rates of process streams or heating exchangers, streams can be pressurized, and/or more trays or stages can be built. These constitute all the independent variables characterizing the process.

Hanson's "description rule" states:

The number of degrees of freedom = The number of independent variables affecting separation that can be arbitrarily set by construction or operation (directly controllable variables)

The use of the rule is quite simple, but some of the terms (independent variables, affecting separation, arbitrarily

set, directly controllable) need further clarification, best accomplished by some detailed examples.

EXAMPLE: ABSORPTION COLUMN

How many variables must be set to completely describe the operation in Figure 1? Visualize operating this column and count up what factors can be controlled. Let C_1 and C_2 be the number of components in each feed, respectively, and let N be the number of stages:

controllable variables	#
1. Feed flow rates	2
2. Feed compositions	$(C_1 - 1) + (C_2 - 1)$
3. Feed temperatures	2
4. Feed pressures	2
5. Stage pressures	N
6. Heat loss from stages	N
7. Number of stages	1
TOTAL	$C_1 + C_2 + 2N + 5$

Thus, according to the description rule, in order to completely define this separation process, one must specify $(C_1 + C_2 + 2N + 5)$ independent variables. Note that there is a free choice as to *which* variables will be set for a desired problem definition. The description rule tells us only *how many* variables must be set.

This example probably requires additional explanation to make the method clear. Item (1) presumes there is a valve on each of the entering streams and that altering these flow rates will, in fact, *affect the separation*. Accepting this latter point requires developing some physical insight or intuition, which is probably the most demanding requirement of this method. Can you see how changing any of the seven items would alter the separations achieved? Note that our analysis did *not* consider that there also might be valves on the *outlet* streams, since they could *not* be *arbitrarily* controlled. What would happen if an outlet valve were closed?

Item (2) presumes direct control of the compositions of each feed, noting that only $(C - 1)$ composition variables are *independent* in a mixture of C components. A simpler, alternate way to count the first two items is to lump them as "feed flow and composition," for a total of $(C_1 + C_2)$ variables. This is like visualizing a manifold of pipes, each carrying pure component, mixing together to form the feed. Note each pipe in the manifold can have a valve, which may be arbitrarily turned to adjust flow rate (Figure 2).

Item (3) presumes there are heat exchangers on the entering feeds. There would not normally be heat exchangers on the outlet streams. The "equilibrium stage" concept tells us that the two streams leaving any stage are "saturated," i.e., in equilibrium with each other. Thus,

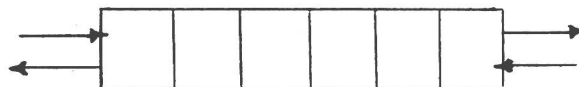
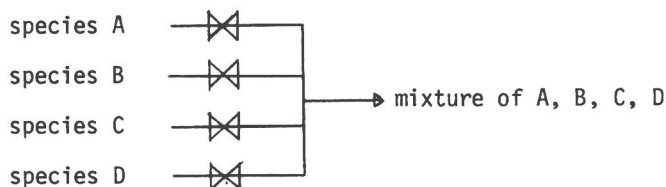


Figure 1.



(4 valves; therefore 4 controllable variables)

Figure 2.

the outlet stream temperature is *not directly* controllable. It is a *dependent*, rather than independent, variable. It is vital that engineers develop a feel for what can be *directly* controlled by operation or construction, since the description rule *only* counts *independent* variables. If operating this column, how could one indirectly change the outlet stream temperature?

Item (4) presumes that the feed pressures can be altered, for example, by manipulating the pressure drop across the feed valves with pumps.

Item (5) similarly presumes one may pressurize each stage separately. Normally columns are designed to minimize pressure drop throughout. In such cases, a *column* pressure is specified, which sets all N of these pressure variables.

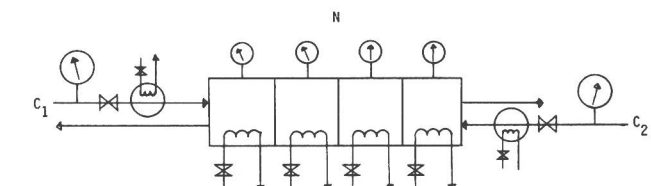
Item (6) essentially presumes a heat exchanger at each stage. In a typical column, heat losses might be controlled by adding or removing insulation. One usually wants to design a column with *no* heat losses (adiabatic), which specifies all N of the heat loss variables as being zero. Or one might design an isothermal column, which is another way to specify these N variables.

Item (7) is the first variable considered here that is associated with *construction*, all the others being related to *operation*. Note that drawing a picture of the column is tantamount to constructing it. There is only one construction variable here which affects separation: the number of equilibrium stages built. Students may say "Wait a minute! I have a lot of construction choices, such as column diameter, spacing between stages, column wall thickness, construction material, stage design, etc." The crucial question is: Do these *affect separation*? Column diameter and spacing between stages will only affect column capacity, i.e., what flows it can handle, but not what separation is achieved. While wall thickness or construction material may have some effect on separation in terms of altering column heat losses, note that column heat losses have already been counted as directly controllable and cannot be counted again. One must be on guard to avoid such redundant or double-counting when applying the description rule. The stage design does not enter as a variable, since this module will only consider equilibrium stages. The correction for non-ideal stages (stage efficiencies) is normally added *after* the ideal case is solved.

It is helpful to modify the drawing of the process to provide a record of the presumptions made in counting controllable variables as shown in Figure 3.

SELECTING REPLACEMENT VARIABLES: PROBLEM DEFINITION

The exercise in Figure 3 is meaningless until the results obtained are applied. *How many* variables must



with the following symbol meanings

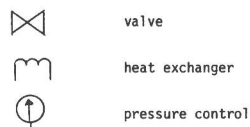


Figure 3.

be set is now known, but there is a free choice as to *what* independent variables will be set to define the problem. A practical limitation forces us to select those variables which make the problem solution less difficult. Normally, the variables describing the feeds (flow rate, composition, temperature, pressure), the column pressure and the heat loss must be set. Note that this sets all but one of the available number of variables.

If the problem at hand is a "design problem," the number of stages required to effect the desired separation need to be calculated. The description rule tells us that only *one* separation variable may be set independently. In essence, this is *replacing* a variable in the controllable variable list with another independent variable. This might be a percent recovery, a composition in a stream, etc. Once this value is chosen, the problem is completely defined, and it is *possible* (not necessarily easy!) to solve for all the dependent variables or unknowns.

Conversely, an "existing column problem" requires determining the separation achieved in a given column. In this case, the one remaining variable is set by stating the number of stages in the column. Here our replacement variables happen to be the same as the list of controllable variables; however, do not confuse the controllable list with the replacement list, the former is just a *tool* to assure the latter properly defines a problem.

Another possible problem might ask for the minimum flow ratio of feeds. Let us tabulate a replacement variable list for such a problem:

replacement variables	#
Feed compositions	$(C_1 - 1) + (C_2 - 1)$
Feed temperatures	2
Feed and column pressure	$2 + N$
Column heat loss	N
Number of stages	1
Separation desired	1
Flow rate of one feed	1
TOTAL	$C_1 + C_2 + 2N + 5$

Note that minimum flow implies the number of stages needed to effect the separation is infinite, which sets this variable. Can you formulate another problem definition and construct the appropriate replacement variable list for the absorption column example?

EXAMPLE: DISTILLATION COLUMN

Is the following problem properly defined?

The ordinary distillation column shown in Figure 4 has 30 stages total, with 10 above the feed. The feed composition is 10% *A*, 30% *B*, 40% *C* and 20% *D*. The feed enters subcooled by 10°C at the column pressure of 3 MPa. It is desired to recover in the top product 90% of the *B* fed. What reflux ratio is required?

Do not solve this problem, but see if there is a solution. Let us apply the description rule:

controllable variables	#
1. Feed composition	3
2. Valves (feed, reflux)	2
3. Heat exchangers (feed, condenser, reboiler, stages)	33
4. Pressures (feed, stages)	31
5. Stages (top and bottom sections)	2
TOTAL	71

(Indicate on Figure 4 what presumptions were made here.)

Let us compare this with the variable list set by the problem statement:

replacement variables	#
1. Feed composition	3
2. Pressures (feed, stages)	31
3. Thermal condition of feed	1
4. Total number of stages and feed location	2
5. Separation specification	1
TOTAL	38

What's wrong?

It is typical in problems of this type that certain things are tacitly understood. Presuming that the column was intended to be well insulated, this sets thirty more variables, namely the heat loss on each stage is zero. This still apparently leaves us short three variables (71 - 68). Actually, the fact that the problem asks only for a flow *ratio* (reflux/top product) means that an *arbitrary flow rate* can be chosen. For example, a top product rate of unity could be picked. (Note: without setting this flow information, the problem is not completely defined in the

sense that nothing is known about the column capacity.) So, there are still two missing variables and this problem is underspecified. What else would be reasonable to set?

While the problem only asks for reflux ratio, note that if it can be solved then *all* other unknowns can be found also. For example, suppose one wanted to know the condenser and reboiler duties. (Note this would actually be a ratio based on the arbitrary flow rate chosen earlier, e.g. heat duty per unit top product.) Since there is a partial reboiler (both vapor and liquid leave), the thermal condition of the bottom product is known to be saturated. But what about the top product? Is it saturated or subcooled leaving the "total condenser" (only liquid leaves)? Clearly, it would be impossible to find the condenser duty without information on the thermal condition of the top product and so this variable must be set.

This leaves one final variable to be defined. This might be another variable defining the separation, such as recovery of *C* in the bottom product, the composition of *B* there, etc. The main caution is that it must be an *independent* variable. Could one set the recovery of *B* in the bottom product? Also, the value set must lie within the limiting range of physical possibility. For example, if *A* is more volatile than *B*, the recovery of *A* in the top product cannot be set as less than the recovery of *B* there. With these cautions, the properly defined problem is summarized:

replacement variables	#
1. Composition of feed	3
2. Pressures (feed, stages)	31
3. Thermal condition of feed	1
4. Total number of stages and feed location	2
5. Insulated column	30
6. Distillate flow rate	1
7. Thermal condition of top product	1
8. Separation specifications	2
TOTAL	71

Note the important fact that in designing an ordinary distillation column *only two* separation specifications may be arbitrarily chosen.

A final hint: it is extremely useful to put the controllable and replacement lists side by side. This helps one to ensure the same assumptions are used to assign the variables as were to count them.

LITERATURE CITED

- Hanson, D. N., J. H. Duffin and G. F. Somerville, "Computation of Multistage Separation Processes," Chapter 1, Reinhold, NY (1962).
- C. J. King, "Separation Processes," Chapters 2 and 4, McGraw-Hill, NY (1971).

STUDY PROBLEMS

- In the absorption column example, why is the number of stages counted as a controllable variable, but not the number of components in each feed or their identities?

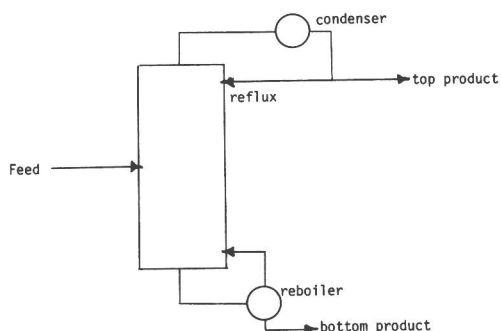


Figure 4.

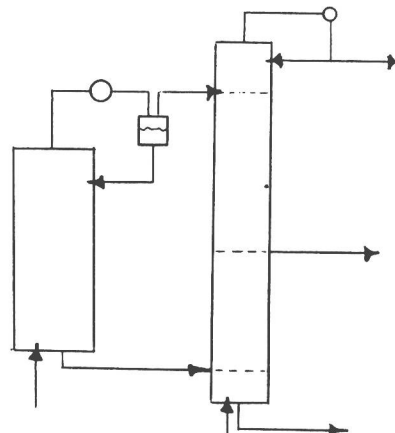
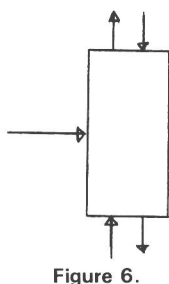
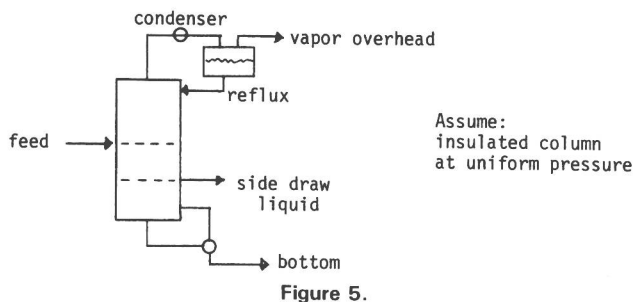
Table 1.

controllable variables	#	design problem replacement variables	#
1) Feed composition	$C - 1$	1) Feed composition	$C - 1$
2) Valves (feed, reflux)	2	2) Flow rates (feed, reflux, side draw)	3
3) Heat exchangers (feed, condenser, reboiler)	3	3) Thermal conditions (feed and reflux)	2
4) Pressures (feed, column)	2	4) Pressures (feed, column)	2
5) Number of stages in each section	3	5) Feed and side draw location	2
6) Feed and side draw location	2	6) Recovery in overhead and bottom	2
		7) Side draw composition	1
	$C + 11$		$C + 11$

2. In the distillation column example:
 - a) Why are only three replacement variables listed for feed composition when four values are given in the problem statement?
 - b) Is it possible to consider a valve on the bottom product stream in counting controllable variables?
 - c) Comment on changing Item (5) in the controllable variable list to: number of total stages and feed location.
 - d) Comment on changing Item (7) in the replacement variable list to: condenser duty.
 - e) Comment on adding to the replacement variable list: relative volatilities of components.
 3. Contrast the difficulty of designing an ordinary distillation column for a binary (two-component) feed versus for a multicomponent feed.
 4. Identify the errors in Table 1, a degrees of freedom analysis for the column shown in Figure 5.
- b) List the variables which need to be set to design this column.
 - c) List the variables which need to be set to determine the separation in an existing column.
 2. Compare the information needed in McCabe-Thiele analysis with the degrees of freedom for an ordinary distillation column.
 3. List the variables which must be set to calculate the minimum stage requirement of an ordinary distillation column. Compare your list with the information needed in the Fenske minimum stages equation.
 4. Repeat Homework Problem 3 for the case of minimum reflux and the Underwood minimum reflux equation.
 5. The Gilliland correlation referred to in many texts on distillation (e.g. Reference 2) relates the number of stages N in an ordinary distillation column to the values of the actual and minimum reflux ratio, R and R_m respectively, and to the minimum number of stages N_m for the desired separation: $N = f(R, R_m, N_m)$. Can such a correlation be generally valid?
 6. Is the following the correct amount of information provided to solve the system in Figure 7? If not, indicate how it should be modified. The columns in Figure 7 operate adiabatically at 100 MPa. The first column contains seven ideal stages. The vapor feed is

HOMEWORK PROBLEMS

1. a) How many degrees of freedom are there for the process indicated in Figure 6?



at 100 MPa and is 20°C superheated with analysis:

species	nC_4	iC_4	nC_5
mol/min	50	70	40

80% of the iC_4 is to be recovered in the overhead vapor. The feeds to the second column are located optimally. Reflux in the second column is 10% greater than minimum and is 5°C subcooled. The saturated vapor feed to

the second column is at 100 MPa with analysis:

species	C_3	nC_4	iC_5
mol/min	30	60	20

90% of the nC_5 is to be recovered in the bottoms and 90% of the iC_5 in the top. 40 mol/min of side draw vapor is to be removed, containing 25% C_3 . Find the reflux rate in column 1 and the number of stages and side draw location in column 2.

Degrees of Freedom by the Design Variable Method

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OBJECTIVES

Upon completing this module, the student should have a clear understanding of the relationship between design and degrees of freedom of staged separation processes, and be able to determine the latter by the design variable method. As a result of this knowledge, the student should be able to:

1. Define and identify:
 - a) degrees of freedom
 - b) variable (design, intensive, extensive, repetition)
 - c) restriction (energy balance, material balance, inherent, and additional due to combination of elements)
 - d) elements
 - e) units
2. Determine the degrees of freedom of simple process elements from first principles.
3. Resolve process units into constitutive elements and combine the degrees of freedom of the individual elements to yield the degrees of freedom of entire units.

PREREQUISITE MATHEMATICAL SKILLS

1. Elementary algebra.

PREREQUISITE ENGINEERING AND SCIENCE SKILLS

1. Material and energy balances.
2. The Gibbs phase rule.

to resolve a complex process into simple process elements and combine the degrees of freedom of the constituent elements to calculate the degrees of freedom of the entire unit.

DESIGN AND DEGREES OF FREEDOM

Consider the design of a storage tank for an ideal gas. If the givens are the quantity of gas and the temperature and pressure at which the gas is to be stored, then the volume of the tank is uniquely determined by the ideal gas law; i.e., the system is said to have no degrees of freedom. On the other hand, if the pressure is not given, the system is said to have one degree of freedom and one independent variable (the storage pressure) must be specified in order to determine the tank volume.

Although this is completely obvious in the case of the simple tank, consider the design of a multicomponent distillation column. How many variables must be specified such that a particular yield and purity of distillate is obtained? One cannot independently specify all the variables, even if one could successfully identify them all, because some are interrelated via mass and energy balances and equilibrium phase relations. And if too few are specified, an undesired product yield and purity may result. In this case, the correct number of variables which must be specified cannot be intuitively determined, as in the storage tank.

Fortunately, however, the exact same procedure can be used to determine the degrees of freedom for the storage tank and the multicomponent distillation column, as well as for any equilibrium stage process. This procedure, simply stated, is:

- a) Count the system variables.
- b) Count the restricting relationships.
- c) The difference between (a) and (b) is the degrees of freedom.

For extremely simple systems, such as the storage tank, this procedure is almost intuitive, while for more complex systems, a self-consistent systematic procedure must be followed to insure that all the variables and restrictions have been properly accounted for. One such procedure is the design variable method.

INTRODUCTION

The purpose of this module is to teach readers the design variable method for determining the degrees of freedom of staged separation processes, and perhaps of more fundamental importance, the translation of degrees of freedom into appropriate specifications of design variables to completely define a process design. At the completion of this material, students should be able to enumerate the variables and restrictions of simple process elements from first principles. They should also be able

PRINCIPLES OF THE METHOD

The design variable method is simply a self-consistent system of classifying and accounting for variables and restrictions based upon the Gibbs phase rule, the first law of thermodynamics, and the law of conservation of mass. The difference between the number of variables and restrictions represents the degrees of freedom or the number of independent variables of the system, N_i . This statement may be represented mathematically as

$$N_i = N_v - N_c \quad (1)$$

where N_v is the total number of system variables and N_c is the total number of restrictions. What are these variables and restrictions?

Variables

The Gibbs phase rule gives the degrees of freedom of any system in thermodynamic equilibrium:

$$N^o = C + 2 - P \quad (2)$$

where N^o is the degrees of freedom of an equilibrium system, C is the number of components, and P is the number of phases present at equilibrium. N^o also represents the number of intensive variables such as concentration, temperature, pressure, etc., determined solely by the state of the system and completely independent of the absolute or relative quantity of material. Quantity-dependent variables are termed extensive variables, e.g., the flow rates of material and energy streams. The sum of the intensive and extensive variables represents the total number of variables of the system, N_i .

Since the phase rule is based on thermodynamic equilibrium, the design variable method, as presented here, is strictly applicable only to equilibrium systems, staged equilibrium processes, or systems reducible to staged equilibrium processes.

Restrictions

A restriction is defined as a relationship between two or more variables. Although the enumeration of system variables is quite straightforward, determination of the number of restrictions, N_c , is more difficult due to ease of omission or duplication. Thus, it is necessary to follow some arbitrary, but consistent procedures, which will yield the correct number. For this purpose, restricting relationships will be classified as energy balance, material balance, and inherent restrictions.

Energy Balance Restrictions

The first law of thermodynamics generally states that the total energy entering a system is equal to that leaving plus any accumulation within the system. For flow processes, the first law usually simplifies to an enthalpy or heat balance. An energy balance is equal to one restriction ($N_c = 1$).

Material Balance Restrictions

A flow system must also be in material balance according to the law of conservation of mass. An over-all

material balance can be written for each of the C components present, or one can write one over-all mass balance and $C - 1$ independent component balances. In either case, material balance restrictions represent a total of $N_c = C$ restrictions.

Inherent Restrictions

Certain restrictions are inherent to the particular system under consideration. These restrictions usually take the form of identities between variables. The ideal gas law may be classified as an inherent restriction in the storage tank design problem. Equality of variables in entering and exiting streams or in streams joining different elements is another example; e.g., composition, temperature, pressure.

Is it important to always classify a restriction in the same manner? No, A particular restriction can be arbitrarily classified as an inherent, material balance, or energy balance restriction. However, *counting must be performed consistently* in order to avoid omission and redundancy.

In order to understand how the design variable method is applied, let us consider the storage tank design problem again. The system variables are pressure, temperature, quantity of gas, and volume of the tank. These variables are accounted for as follows: The storage tank is a single-component ($C = 1$), single-phase ($P = 1$), equilibrium system, and the number of intensive variables is given by the phase rule: $N^o = 1 + 2 - 1 = 2$. The two intensive variables are, of course, temperature and pressure. To these must be added the number of extensive variables, which in this case is two, the quantity of gas and the volume of the tank. Thus, the total number of variables in the system is $N_v = 2 + 2 = 4$.

Can all four variables be independently specified? No, because they are related via the ideal gas law. Thus the ideal gas law represents an inherent restriction in the system and $N_c = 1$. Therefore, the degrees of freedom for the storage tank are $N_i = 4 - 1 = 3$, and we must independently specify three variables to completely define the design.

Normally, however, some variables are previously specified as a design basis. The difference between the degrees of freedom of the system, N_i , and the number of specified variables, N_s , is the number of design variables, N_d :

$$N_d = N_i - N_s \quad (3)$$

The design variables are the primary ones of interest, since they represent the number of independent variables available to the designer for process specifications. For example, in the storage tank design problem, the quantity of gas to be stored will probably be the design basis. Also, the tank pressure may be dictated by safety and strength of materials constraints. Thus, in this case, $N_s = 2$, $N_d = 3 - 2 = 1$, and only the temperature can be independently specified. Furthermore, if the gas is to be stored at ambient temperature, then $N_s = 3$ and $N_d = 0$. Now there are no design variables and the system is invariant. For the case where there are no specified

Table 1. Elements

Elements	Single Stream	Stream Divider ⁽²⁾	Mixer	Pump Heater or Cooler	Simple Equilibrium Stage	Side Stream Stage	Feed Stage	Total Condenser or Reboiler	Partial Condenser or Reboiler	Simple Separator	Heat Exchanger
<i>Variables</i>											
Single-phase streams:											
intensive	$C+1$	$2(C+1)$	$3(C+1)$	$2(C+1)$	$2(C+1)$	$2(C+1)$	$3(C+1)$	$2(C+1)$	$C+1$	----	$4(C+1)$
N_v^e extensive	1	3	3	2	2	2	3	2	1	----	4
Two-phase streams:											
intensive	----	----	----	----	C	C	C	----	C	$2C$	----
extensive	----	----	----	----	2	3	2	----	2	4	----
Heat streams	----	1	1	1	1	1	1	1	1	1	1
TOTAL	$C+2$	$2C+6$	$3C+7$	$2C+5$	$3C+7$	$3C+8$	$4C+9$	$2C+5$	$2C+5$	$2C+5$	$4C+9$
<i>Restrictions</i>											
N_c^e Material balance	----	C	C	C	C	C	C	C	C	C	$2C^{(3)}$
Energy balance	----	1	1	1	1	1	1	1	1	1	1
Inherent ⁽¹⁾	----	----	----	----	----	----	----	----	----	----	----
TOTAL	0	$C+1$	$C+1$	$C+1$	$C+1$	$C+1$	$C+1$	$C+1$	$C+1$	$C+1$	$2C+1$
$N_f^e = N_v^e - N_c^e$	$C+2$	$C+5$	$2C+6$	$C+4$	$2C+6$	$2C+7$	$3C+8$	$C+4$	$C+4$	$C+4$	$2C+8$
<i>Typical Specifications</i>											
Feed stream	$C+2$	$C+2$	$2(C+2)$	$C+2$	$2(C+2)$	$2(C+2)$	$3(C+2)$	$C+2$	$C+2$	$C+2$	$2(C+2)$
Pressure	----	1	1	1	1	1	1	1	1	1	1
Heat stream	----	1	1	1	1	1	1	1	1	1	1
Product stream rate	----	1	----	----	----	1	----	----	----	----	----
Product stream temperature	----	----	----	----	----	----	----	----	----	----	1
TOTAL	$C+2$	$C+5$	$2C+6$	$C+4$	$2C+6$	$2C+7$	$3C+8$	$C+4$	$C+4$	$C+4$	$2C+8$

(1) Inherent restrictions arise more frequently in combining elements.

(2) Simple stream dividers are essentially adiabatic, and the feed stream pressure is generally the same as the divider pressure. If these are listed as inherent restrictions, N_f^e would be reduced to $C+3$.

(3) Since there is no mass exchange between streams, there are C material balances per stream.

variables ($N_s = 0$), the number of design variables is synonymous with the degrees of freedom of the system.

ANALYSIS OF ELEMENTS

The detailed enumeration of all the variables and restrictions in a complex equilibrium stage process could become quite tedious and time-consuming. All such processes, however, are combinations of a relatively few simple elements, the degrees of freedom of which can be easily calculated from first principles and catalogued for subsequent use. The degrees of freedom of the unit can then be calculated by combining the degrees of freedom of all the constituent elements and according to some simple rules.

The degrees of freedom of a simple element are given by

$$N_i^e = N_v^e - N_c^e \quad (4)$$

where the superscript e denotes an element. A summary of the analysis of some simple elements according to Equation 1 is presented in Table 1. Schematics of the elements appear in Figure 1. The detailed procedure for determining the degrees of freedom of simple elements from first principles will be illustrated in this section using the simple equilibrium stage, the feed stage, and the side stream stage as examples.

Consider the schematic of the simple equilibrium stage

in Figure 1, in which two feed streams of C components are introduced (e.g., a liquid stream and a vapor stream). The number of intensive variables in each of these streams is given by the phase rule: $N^o = C + 2 - 1 = C + 1$; while the mass flow rate contributes one extensive variable, a total of $C + 2$ per stream. Since the two product streams are in equilibrium, the total number of intensive variables of these two streams together is given by the phase rule: $N^o = C + 2 - 2 = C$. This is because the compositions, temperatures and pressures of the two product streams are not independent, but related by equilibrium relationships (e.g., Raoult's or Henry's law). The flow rate of each stream, however, is independent and contributes one extensive variable for each stream. The rate of heat exchange, or heat stream, between the stage and surroundings contributes one additional extensive variable. The total number of independent variables for the simple equilibrium stage is thus,

$$N_v^e = \underbrace{2(C+2)}_{\text{two feed streams}} + \underbrace{(C+2)}_{\text{two product streams}} + \underbrace{1}_{\text{heat stream}} = 3C + 7.$$

The restricting relationships consist of C material balances and one heat balance, or $N_c^e = C + 1$. The degrees of freedom of the simple equilibrium stage are then

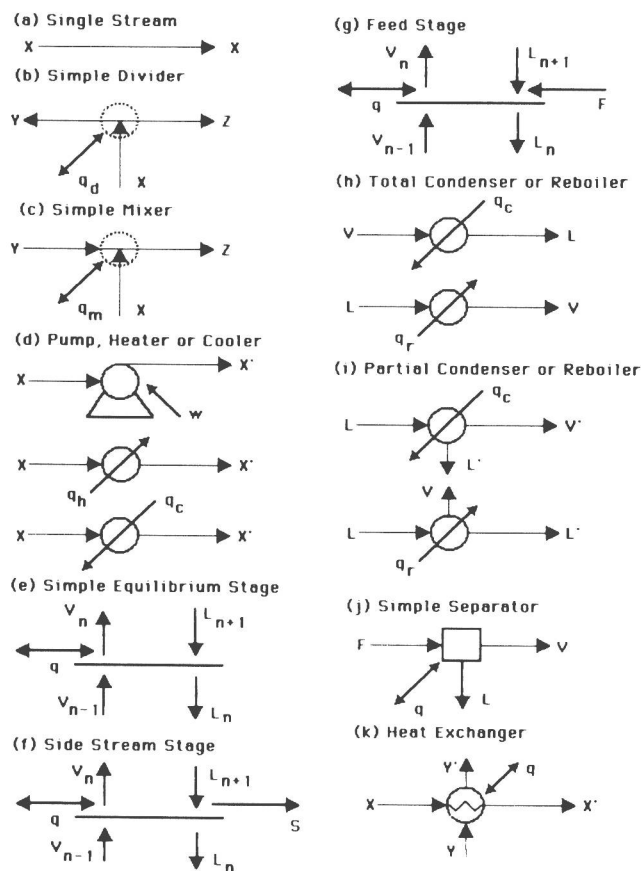


Figure 1. Elements.

given by

$$N_i^e = (3C + 7) - (C + 1) = 2C + 6.$$

A complete specification of the simple equilibrium stage would probably consist of the following:

Specification	N_i^e
Composition of each feed stream	$2(C - 1)$
Temperature of each feed stream	2
Pressure of each feed stream	2
Rate of each feed stream	2
Stage pressure	1
Heat leak (or temperature of the stage)	1
	$2C + 6$

These specifications illustrate two rules which are followed in enumerating or specifying variables. First, the number of variables due to composition are $C - 1$, and not C . This is because the C th composition is provided by an internal mass balance in each stream; i.e., the C th composition is a dependent variable. Second, it will arbitrarily be assumed that entering streams have $C + 2$ variables, which are, in general, independent of the conditions within the element; i.e., the variables are specified before the streams enter the element. However, streams exiting the element are at the temperature and pressure of

the element; i.e., the variables are assigned before the stream leaves the element and before the occurrence of pressure or temperature change.

In counting the intensive variables of the two product streams, it was stated that the temperature and pressure of these two streams could not be independently assigned. However, the temperature and pressure of the stage can be independently assigned, and by the convention just cited, these are identical to the temperature and pressure of the two product streams. Is this a contradiction? No, because now one cannot specify the composition of either product stream as before, since the complete specification of the feed streams and the temperature and pressure of the stage fixes the compositions of the product streams by equilibrium and material balance.

For the feed stage, the number of variables in the simple equilibrium stage is increased by the total number of variables of the additional feed stream, $C + 2$. The restrictions remain the same and therefore,

$$N_i^e = 3C + 7 + (C + 2) - (C + 1) = 3C + 8$$

The feed stage can be specified in the same manner as the simple equilibrium stage ($2C + 6$ variables) with the inclusion of the additional feed stream specification ($C + 2$ variables).

By this time, students should realize that the calculation of degrees of freedom is little more than a mechanical exercise. The value of this exercise lies in the understanding of the interrelationships between variables and restrictions, and in translating the resultant design variables into convenient design specifications. To emphasize this point, let's calculate the degrees of freedom of the side stream stage, using three different methods to show how variables and restrictions are related and sometimes even interchangeable.

Method A

The side stream stage is the same as the simple equilibrium stage, except that an additional product stream is withdrawn (see Figure 1). The side stream can be viewed as being identical to one of the other two product streams and therefore, in equilibrium with the remaining product stream. Thus, the side stream can be combined with the other two product streams for the purpose of enumerating intensive variables, as was done for the simple equilibrium stage, $N^o = C + 2 - 2 = C$. The mass flow rates of the three product streams are independent and contribute three extensive variables. The heat stream contributes one additional extensive variable. As before, the two feed streams contribute a total of $2(C + 2)$ variables. The total number of variables is then:

$$N_v^e = \underbrace{(C + 3)}_{\text{three product streams}} + \underbrace{2(C + 2)}_{\text{two feed streams}} + \underbrace{1}_{\text{heat stream}} = 3C + 8$$

In this method, the number of restrictions is the same as