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Hanson, Donald N.; Duffin, John H.; Somerville, Graham F.

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COMPUTATION OF MULTISTAGE SEPARATION PROCESSES

Donald N. Hanson
University of California
Berkeley, California

John H. Duffin
U. S. Naval Postgraduate School
Monterey, California

Graham F. Somerville
University of California
`Berkeley, California*

CHAPTER 1. INTRODUCTION

The calculation procedures to be discussed in subsequent sections of this book offer a means of solution to a wide variety of separation problems. The very fact that they are so versatile, however, requires in their utilization a thorough understanding of the formulation or setting up of the separation problems. To set up or describe a separation problem to be calculated is simply to assign values to the number of independent variables needed to define the problem. The calculation then yields the values of all the dependent variables.

Although it is quite as important as the calculation, the subject of problem description has, until recently, been neglected in distillation literature. Gilliland and Reed,[2]* and Kwauk[5] have presented papers giving a rigorous treatment of problem description and Robinson and Gilliland[6] discuss the subject briefly in their book. The great bulk of the literature has been concerned with development of calculation methods suitable to the design of columns, and the problem set up to test the methods has almost inevitably been the same, viz., the independent variables have been two separation specifications, the reflux or reflux ratio, and the stipulation that the feed plate was to be located at the optimum point. Here, then, .calculations are made primarily to obtain values for the number of enriching and stripping stages, although they also yield the separation that will result for each component of the feed. This problem has been illustrated so often that its formulation is intuitive to those working in distillation design. However, the simple, general rule which governs how many variables are independent and must be set to define a separation problem, is not as well known. Therefore, it becomes desirable to state and discuss the rule. The rule as discussed here is, in essence, the same as the more elegant formulations of Kwauk but is much easier and quicker to apply.

Associated with any fractionation problem are a number of variables which describe the column and its operation. These variables are flows,

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*Superscript numbers refer to bibliographical references at end of book.

temperatures, separation specifications, number of stages, column diameter, and in general, any quantity that satisfies the requirement of describing the column or its operation. Obviously, not all of these variables are independent and can be arbitrarily set, and the *number* of independent ones changes with the column used and the operation performed. A simple, general problem-description rule called the Description Rule, which will hold for any multistage contacting unit, can be stated as follows: *To completely describe the separation operation, the number of independent variables which must be set must equal the number that can be set by construction or controlled by external means.* Setting a lesser number in the problem description yields an infinitude of answers in solving for values of the remaining variables, whereas setting a greater number may lead to no answer.

The use of the rule is extremely simple, but the terms "variables which can be set by construction or controlled by external means" are unfortunately vague and can best be clarified by the use of examples. A simple distillation column is shown in (a) of Figure 1. The act of drawing the column and other equipment is tantamount to construction, and and feature of the drawing has been set by construction and hence is an independent variable. Not all the features of the column, however, enter the fractionation equations, and so these extraneous features can be neglected. The important independent variables that can be set by construction in the figure are the numbers of theoretical stages in each column section. The fractionation equations written must, of course, recognize such structural stipulations as a partial condenser, but the influence of the bulk of the structural details on the fractionation can at present be only implicitly expressed in the estimate of the over-all stage efficiency for the column.

In addition to being able to construct column (a) of Figure 1 with any desired number of theoretical stages in the two sections, there are a number of variables which can be externally controlled and determined by simply looking at the figure. It is apparent that an arbitrary amount of feed material may be introduced into the column, that an arbitrary amount of heat may be introduced in the reboiler, and that (within limits) an arbitrary amount of energy or heat may be withdrawn at the condenser. These are, then, additional independent variables. Lastly, it is apparent that the pressure at some point in the column can be arbitrarily set and controlled, and that the feed may have any composition and enthalpy content.

The total list of independent variables which must be fixed to completely describe the problem is shown in the following table:

Independent Variables	Number
Feed composition (N components)	$N-1$
Feed amount	1
Feed enthalpy	1
Pressure	1
Condenser duty	1
Reboiler duty	1
Theoretical enriching stages	1
Theoretical stripping stages	1
	$N + 6$

The fractionation problem with the $N + 6$ specific independent variables above would seldom be defined. Among the variables listed, the condenser and reboiler duties are generally of little interest as independent variables. Instead, it is usually desired to specify something about the separation. This can be done, since for this example, all of the listed independent variables can be replaced in describing the problem with an equal number of any other independent variables; variables may be chosen which are most pertinent to the problem at hand. The Description Rule serves to tell how many independent variables must be set. The ones used are a matter of free choice.
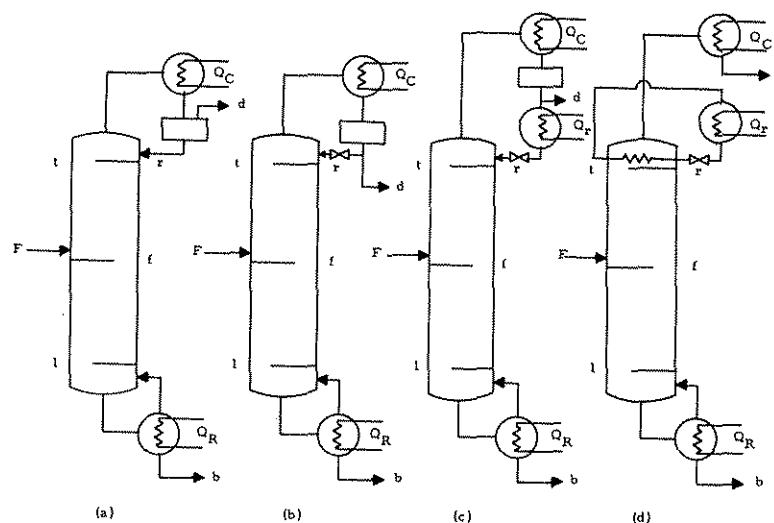

Figure 1.

Practical limitations enter, however, in the selection of independent variables, because the calculations are too difficult to carry out unless certain variables are set. Thus it is imperative that variables describing the feed, and also the column pressure, be set in order to do calculations, and it becomes convenient in thinking about the Description Rule to consider these as always set. For column (a) of Figure 1, the last four variables of the above list are available for replacement by other variables in a specific problem description.

The Description Rule is just as easily applied to more complex operations. As a further example, column (a) of Figure 2 shows a distillation column with a side stream, S, withdrawn from a stage in the enriching section. Again, the column may be fed with feed of any amount, com-

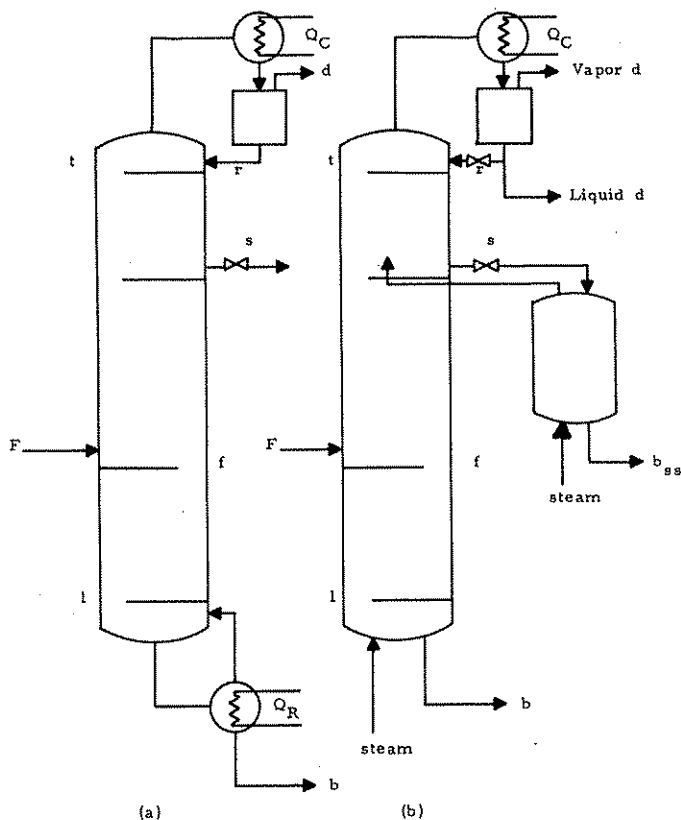
(a)   (b)

Figure 2.

position, and enthalpy, and may be operated at any pressure; these independent variables can be considered as arbitrarily set. From inspection of column (a), Figure 2, the remaining independent variables are:

Condenser duty	1
Reboiler duty	1
Enriching stages	1
Intermediate stages	1
(between side-draw stage and feed stage)	
Stripping stages	1
Side-draw amount	1
$\sum = 6$	

Thus there are six independent variables (in addition to feed variables and column pressure) compared to four for the column and operation of (a) in Figure 1.

Probably the most complex column and operation that will normally be encountered is a crude-oil distillation column, a somewhat simplified illustration of which is shown in (b) of Figure 2. Again, all the feed variables and the column pressure are set, and the remaining variables are written down by simply inspecting the figure and noting the points that may be controlled.

Main stripping-steam amount	1
Main stripping-steam enthalpy	1
Side-stripper-steam amount	1
Side-stripper-steam enthalpy	1
Enriching stages	1
Intermediate stages	1
Stripping stages	1
Side-stripper stages	1
Side-draw amount	1
Condenser duty	1
Reflux amount	1
$\sum = 11$	

The Description Rule may be applied with ease to any multistage operation such as distillation, absorption, extraction, etc., and will yield the number of independent variables that must be set in defining a problem. However, the variables listed in the process of applying the rule are those which are fixed either through construction or through direct external control; and as noted above, these are not necessarily of greatest interest in the definition of a specific problem.

In almost all problems the variables describing the feed and column pressure are set; but when the remaining variables are replaced with other independent variables, four considerations enter:

(a) The replacement variables must be independent.

(b) The variables listed in the problem description must be set at values which lie within the limiting range of possible values.

(c) In some problems, all of the variables counted by the Description Rule cannot be replaced with variables describing the separation.

(d) Those variables should be chosen which allow a reasonably easy solution of the problem.

Each of these considerations needs further discussion.

Variables are independent if they are not defined through a mass balance, an energy balance, or a fractionation equation defining the relation between concentrations, stages, and flows. Column (a) of Figure 1 serves to illustrate these points. Consider the column operating to separate a given amount of feed (an N-component mixture of specified composition and enthalpy) into a top product, d, and a bottom product, b. It is apparent that the amount of component 1 in the top product, $d(x_1)_d$, and the amount of the same component in the bottom product, $b(x_1)_b$, are not both independent, since

$$F(X_1)_F = d(x_1)_d + b(x_1)_b$$

However, one of the two, say $d(x_1)_d$, is independent. In the same way, for other components, $d(x_2)_d$, $d(x_3)_d$, and $d(x_4)_d$ are independent variables, since they are not connected through mass balances. It might be assumed that the amounts of all other components in the top product are also independent, but in this case the Description Rule allows only four independent variables; the remaining variables are thus dependent through fractionation relations.

Variables may also be dependent through energy balance. This can be determined for specific variables only by writing the appropriate energy balance. However, because composition, amount, and temperature are needed to define an enthalpy flow, and since it is uncommon to set all these variables, this is seldom of concern. To illustrate again with (a) of Figure 1, it is possible (although it seems at first glance unlikely), to arbitrarily set the temperatures of any four stages, since they are all independent variables. However, such a fractionation-problem definition would be unusual to say the least.

The second consideration is often of concern because it is not always easy to find the limits within which certain variables can be set. Again, column (a) of Figure 1, separating an N-component mixture, serves as an illustration. For purposes of this illustration, a useful variable for describing the separation of a specified component is introduced, viz., the recovery fraction of that component in a product. The recovery fraction of component i in the top product is defined as the fraction of the amount of that component in the feed which appears in the top product, and therefore for component i this recovery fraction becomes $d(x_i)_d/F(X_i)_F$ and is assigned the symbol $(/i)_d$. The recovery fraction of a component in any other product, in this case the bottom product, is defined in a similar manner: $(/i)_b = b(x_i)_b/F(X_i)_F$. If (as here) there are only two products, then $(/i)_d + (/i)_b = 1$, and only one of these variables is independent.

A set of independent variables describing the problem might then be $(/1)_d$, $(/2)_d$, $(/3)_d$, and reflux amount. Although these can all be set arbitrarily—that is, they can take on an infinitude of values—there are certain limitations on these values. If the vapor-liquid equilibria for the system of components are not highly nonideal and components 1, 2, and 3 are progressively less volatile, it is apparent that $(/1)_d$ must be greater than $(/2)_d$, which in turn must be greater than $(/3)_d$. If $(/1)_d$ is set first, it is limited only to values between 0 and 1. If $(/2)_d$ is set next, it is limited to values between $(/1)_d$ and 0. If then reflux quantity is set, it is limited to values between the minimum necessary to effect the specified separation on components 1 and 2, and infinite reflux. If reflux is set at some value within these limits, $(/3)_d$ is now limited to values within those obtained with either a maximum number of enriching stages or a maximum number of stripping stages (usually an infinity of stages in either section). These limits could all be obtained by calculation; and if a separation problem were to be described by the four variables used above, it would be necessary that the values chosen were within the allowed limits.

The third consideration states that although all the variables obtained through the Description Rule are independent, they are not necessarily all replaceable with a completely free choice of other independent variables. This can best be shown by considering columns (a), (b), and (c) of Figure 1, for which it will be understood that all feed variables and pressure have been set. The only difference between these columns is that column (a) has a partial condenser, (b) a total condenser, and (c) a total condenser plus a reflux cooler. The Description Rule will correctly predict the number of remaining independent variables that must be set to define a problem for each of these columns. These variables are shown in Table I.

Regarding column (a), all the variables listed in Table I have direct bearing on the fractionation being done, i.e., if any one of the variables is changed, the separation will be changed. If the feed contains four components (1, 2, 3, 4, or more), then each of the variables listed could be replaced with the recovery fraction of one of the components or with variables that are functions of concentrations, such as four stage temperatures. Thus, in theory at least, it would be possible to design a column like (a) of Figure 1 to perform a set separation on four components.

TABLE I. INDEPENDENT VARIABLES FROM DESCRIPTION RULE
(in addition to feed variables and column pressure)

Figure 1, Col. (a)	Figure 1, Col. (b)	Figure 1, Col. (c)
Enriching stages	Enriching stages	Enriching stages
Stripping stages	Stripping stages	Stripping stages
Reboiler duty, Q_R	Reboiler duty, Q_R	Reboiler duty, Q_R
Condenser duty, Q_C	Condenser duty, Q_C	Condenser duty, Q_{C1}
	Reflux amount, r	Reflux amount, r
		Reflux cooler duty, Q_r

With column (b), it might be expected that five independent recovery fractions could be set, and (in like manner) six for column (c). However, it is possible to represent (b) and (c) of Figure 1 by the column shown in (d). In this last column the reflux stream has been separated from the top-product stream. In reality this is also occurring with the reflux streams of (b) and (c), since they can be considered as following a closed cycle about the column tops. In all three cases, then, the reflux streams have the same effect in producing liquid flow off the top stage. Also, in column (d), the top product has been shown as condensing in a separate condenser. Thus, if the top product and reflux leave their condensers at the same temperature, column (d) becomes equivalent to column (b); and if they leave at different temperatures, (d) is equivalent to (c). It is apparent that the top-product condenser of column (d) has no influence on the fractionation, since it is completely removed from it. Also, it is apparent that the amount of reflux flow around the closed loop of (d) is irrelevant to the fractionation. The only relevant variable is the heat extracted at the top stage; this can be accomplished with an infinitude of external reflux flows, depending on subcooling. Hence, this heat load can be replaced in both columns (b) and (c) by a separation variable or—what is the same thing—the condenser load of which it is a part can be replaced. Alternatively, the variables associated with the condenser might be taken as reflux amount and reflux temperature, in which case one or the other, but not both, could be replaced by a fractionation variable. In all three cases then (columns (a), (b), and (c)), only four separation variables can replace the variables counted by the Description Rule.

With columns (b) and (c), if the reflux amount were set, it would still be necessary to set the reflux temperature in the problem description. In column (c), in addition to the reflux temperature out of the condenser, the reflux temperature from the cooler would also have to be set. It is usually desirable to set the temperature of the reflux rather than the condenser or cooler duty.

The fourth consideration, that the variables chosen must be such that a reasonably easy solution can be obtained, is the most important. The complete set of equations expressing mass balance, energy balance, and interphase equilibria can be written for any multistage process of fixed numbers of stages operating on any number of components. In general, however, since the equations are nonlinear, no simple solution of the set of equations can be obtained.

If, however, a complete set of flows and temperatures is assumed and equilibrium stages are postulated, the equations can be reduced to a linear set of equations in concentration of the individual components. These equations can be solved to yield compositions in the stages; and from these compositions, corrections to the assumed flows and temperatures can be obtained. The solution of the problem can thus be made into an iterative process converging on the correct solution to the nonlinear set of equations.

In further discussion of the general method of solution, it might be best to direct emphasis to the type of column solution to be discussed in subsequent sections of this work. The problems to be discussed are those for which a column already exists or for which column design is to be effected by calculating the flow requirements of a series of likely columns and choosing the optimum column. It is thus the reverse of the design procedure which calculates the column required at some reflux arbitrarily greater than minimum. Rather, the calculation yields reflux requirements for a column arbitrarily greater than the minimum number of stages at total reflux.

In the setting up of fractionation problems of this type the numbers of theoretical stages in each column section are always independent variables and are set in the problem description. All feed variables and column pressure must also be set. The number of "remaining" independent variables is usually small, and often zero. For column (a) of Figure 1 there are only two; in more complex columns there are still relatively few remaining.

As noted above, the iterative solution of the problem requires the knowledge of all flows and temperatures either through setting as independent variables or through assumption and subsequent correction. Correction procedures for estimation of new values of the assumed variables have been developed only for a few simple problem descriptions, namely the problems in which the remaining variables are chosen either as flows or energy inputs or outputs. Since, in general, it is desirable to work with flows, the remaining variables should be chosen as flows.

As an example, consider the simple distillation column (a) in Figure 1. The particular problem of interest might consist of setting values for the variables:

Enriching stages,
Stripping stages,

Recovery fraction of component 1 in the top product, and
Recovery fraction of component 2 in the top product.

Since this problem cannot be solved directly by the iterative schemes proposed, the solution must be obtained through the solution of a series of simple problems in which the last two variables are replaced with:

Top product amount, and
Reflux amount,

until the values of top product and reflux are found under which the separations obtained on components 1 and 2 are those desired.

It may appear that the method of approach is unnecessarily devious for .what is a rather simple problem. But it is readily applicable, no matter what the last two variables in the problem are. For example, they could be the recovery and purity of a component in a product, or the octane number and amount of a product, or any two variables which are functions of the compositions and amounts of the products. Also, the basic problem is almost as easily solved for complex processes, such as multiple-feeds, interlinked columns, etc., as for simple columns. The real usefulness of the methods then is in their general applicability.

In any multistage separation process, both mass balance and energy balance must be satisfied. However, it is possible by postulating heating or cooling in each stage to create an additional group of independent variables which may be arbitrarily set and held. The number of these variables is equal to the number of stages which are heated or cooled.

Thus, in a distillation process, one of the flows from each stage can be set. This fixes the complete flow map of the column, assuming that reboiler load and condenser load have been replaced in the Description Rule with two flows. Because of the additional independent variables available, a temperature map and composition map can be calculated which are correct for the fixed flow map. In general, this calculation requires the assumption of a temperature map. The set of equations is solved to yield a composition map which is used to correct the temperatures for subsequent iterations.

The postulate of heating or cooling on each stage can be dropped after the converged temperature gradient is found and the flows corrected to correspond to energy balance. The temperature map will usually have to be corrected again, followed by correction of flows, correction of temperatures, etc., until convergence is reached on both. In general, it is not necessary to converge the temperature map before correcting the flow map; but if desired, the temperature map could be converged for any flow map.

Conversely, in a liquid-liquid extraction column, it is advantageous to reverse the order of calculation. A temperature map is set (usually

constant) through the postulate of heating or cooling on each stage. A flow map is assumed, the set of equations is solved to determine compositions, and these compositions are used to correct the flow map for subsequent iterations if desired. The postulate of heating or cooling can then be dropped and the temperature map corrected to correspond to energy balance.