modelling packages in order to identify practical limitations of the simulations at an early stage.

4. Development of separation systems incorporating distillation in order to address specific environmental problems and applications.

Significant applications are expected in the use of *computational fluid dynamics* (*CFD*) *packages* for prediction of effects occurring within distillation equipment. This is a different area of research from the use of the flowsheeting packages and the calculation of equilibrium stages. The CFD approach (generally using commercial packages such as PHOENIX^{TM} and FLUENT^{TM}) has been used to predict single-phase flow patterns (of a vapour phase) from numerical solutions of the Navier–Stokes equation, turbulence equations, and the continuity equation. If the equations of momentum and mass transfer are inserted into the CFD methodology then it may be possible to predict the flow patterns and their effects upon tray performance. However, the major challenge is the consideration and modelling of the three-dimensional froth height and its shape.

Reviews of the state-of-the-art in distillation and the need for and possible directions of future research have been discussed by Fair (1988), Kunesh *et al.* (1995) and Porter (1995). Assessments of advances and developments in distillation equipment regularly appear in the journal literature, e.g. *Chemical Engineering* (*NY*), December 1992; *Hydrocarbon Processing*, February 1989; *The Chemical Engineer* (*IChemE*), September 1987. Fouling and plugging in equipment and a better understanding of the internal flow mechanisms and regimes are areas receiving and requiring further attention, as discussed earlier. Most new ideas tend eventually to become either an academic curiosity, or niche applications, and approximately every 10 years a new technique gains attention and prominence, e.g. reactive distillation, membrane–distillation.

See Colour Plate 38.

See also: **II/Distillation:** Energy Management; Instrumentation and Control Systems; Modelling and Simulation; Theory of Distillation.

Further Reading

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Instrumentation and Control Systems

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Introduction

Distillation columns have been widely used in the past to separate mixtures of liquids into individual components. And even though new separation techniques are being developed, distillation remains the most important separation method applied in the process industries today.

The layout of a simple distillation column is shown in **Figure 1**. A single feed enters the column at the side and two products are produced: the light or most volatile components are withdrawn from the top and heavy components are removed from the bottom. Heat (in the case shown, steam) for evaporation of the liquid is supplied to the reboiler, and heat is removed (in this case, through cooling water) at the top in the condenser. The nomenclature used in this

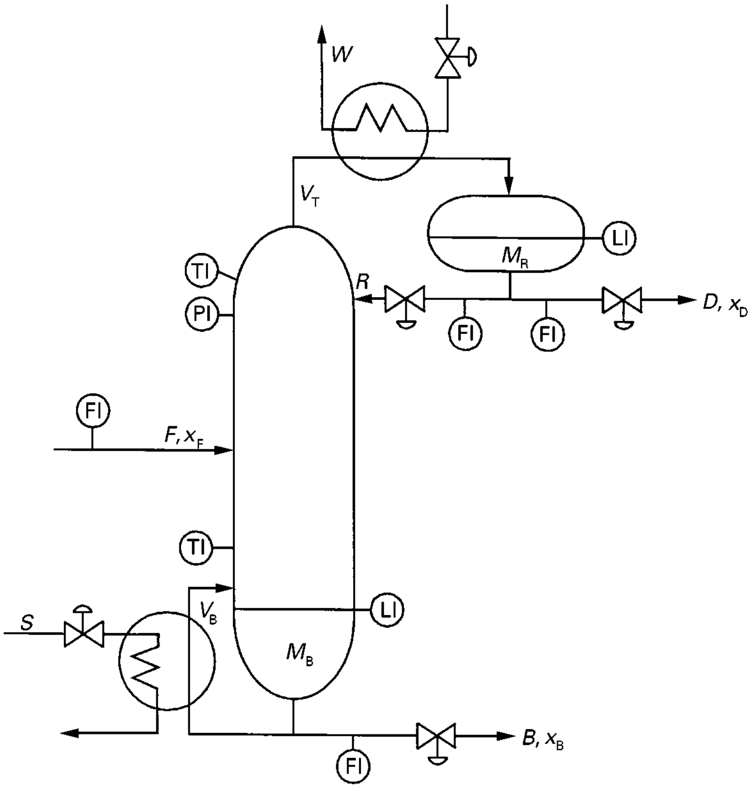

Figure 1 Distillation column layout.

chapter is shown in **Figure 1**, where $V =$ vapour flow, $F =$ feed flow, $R =$ reflux flow, $D =$ distillate flow, $B =$ bottom flow, $x =$ composition, $M =$ mass hold-up and W and S are cooling water and steam flow respectively. In order to maintain constant separation in the distillation column, it should be well instrumented and controlled. The treatment of instrumentation and control techniques will focus primarily on packed columns or columns with trays.

A number of excellent books and review articles have been written about distillation control. Shinskey's book (1984) is a very practical one and provides a good introduction to the subject of distillation control, although a detailed explanation of different approaches to control alternatives is always clear to the novice. The book by Buckley *et al.* (1985) provides a comprehensive treatment of pressure, level and protective controls; control of composition is restricted to a short treatment of composition dynamics in binary columns. The book edited by Luyben (1992) is probably the best starting point: It has been written by several experts in the field of distillation dynamics, instrumentation and control. It provides a comprehensive treatment of distillation models, distillation simulation, identification of distillation processes and selection and comparison of control structures. In addition, several chapters are devoted to particular case studies. The *Process Control Instrument Engineers' Handbook* (Liptak, 1995) also provides three interesting sections on distillation control: one section discusses basic controls, another section discusses advanced controls and in a subsequent section the subject of relative gain calculations is reviewed. The handbook also provides a wealth of information on instrumentation.

A book which gives a good introduction to batch distillation control is the one by Fisher (1990) and Luyben (1992) gives a good overview. Three review articles should be mentioned as a starting point for further reading: the first one is by Tolliver and Waggoner (1980: 195 references), another is written by McAvoy and Yang (1986: 270 references), and the one by Skogestad (1992) also provides easy-to-understand material: it has 206 references.

Because vapour and liquid with a certain energy content are present in the column, basic instrumentation includes measurements of the vapour hold-up (column pressure), liquid hold-ups (column bottom level and reflux drum level) and generally a number of temperatures along the column. In addition, the in- and outgoing flows are usually measured, as shown in **Figure 1**.

Since the feed to the distillation column is often fixed by a preceding process, no control valve is shown in this flow, although sometimes the feed preheater control valve is used to control the amount of feed that vaporizes. This means that five manipulable flows remain and there are essentially five degrees of freedom for control. However, the vapour and liquid hold-ups have to be controlled, which means that $5 - 3 = 2$ degrees of freedom remain, which are generally used for composition control.

Distillation columns pose some interesting control problems. First of all, the process is often highly nonlinear. Secondly, there are five variables to be controlled (pressure, P, level, h_B, level, h_R, composition, x_D, composition, x_B) and five variables which can be manipulated (flows R, D, B, S and W). How the coupling between these controlled and manipulated variables should be established has been an interesting field of study for many years. In our approach a matrix (**Figure 2**) is constructed where all variables are listed; for each combination of controlled output and manipulated input the control quality is determined. The control quality can be established on the basic of speed of control, power of control and the requirement of minimal interaction between control loops. The speed of control is related to the period of oscillation of the control loop at the limit of stability. The shorter this period, the higher the speed of control. The power of control relates to the range over which control is effective. For an acceptable combination, the speed of control should be large, as should the power of control – in other words, the controlled output should respond quickly to changes in the manipulated process input. However, there are not always five variables to be controlled. It could be that there is no strict requirement for the column pressure, in which case it is often optimal to minimize the pressure and open the cooling water valve of the condensor completely. It could also be that there is a strict requirement for the top product composition but no requirement for the bottom composition, in which case there would be an extra degree of freedom.

	← Basic control →				
	P	h_B	h_R	x_D	x_B
W					
B					
D					
R					
S					

Figure 2 Control matrix, showing controlled and manipulated variables.

The issue of column operation, column instrumentation and selection of the right pairing between controlled process outputs and manipulated inputs will be considered in more detail in the following sections.

Objectives for the Separation Process

The main objective of the distillation process is usually the recovery of a valuable component from the feed. In that case there is also often a quality requirement for this valuable component. If the purity of the valuable component is low, then the product has little value. If the concentration meets the specification, then the product value is high.

There may be no distinct quality requirement, in which case the economic value of the product could be a continuous function of the product properties.

Let us assume that both top and bottom product represent an economic value. Then an economic objective for the operation of the process could be defined as:

$$J = c_D D + c_B B - c_F F - c_S S \qquad [1]$$

where c_D is the top product value (\$ kg^{-1}), c_B the bottom product value (\$ kg^{-1}), c_F the cost of the feed (\$ kg^{-1}) and c_S the cost of the steam (\$ kg^{-1}). The use of cooling water usually involves little cost and fixed costs do not play a role in the optimization of the operation.

If F is fixed and the overall material balance $F = B + D$ is substituted in eqn [1], then the variable part J_d of eqn [1] can be written as:

$$J_d = D - \frac{c_S}{c_D - c_S} S \qquad [2]$$

Figure 3 shows both terms as a function of the steam flow to the column reboiler. When S increases, the yield of the valuable product approaches asymptotically a value which is equal to the amount of top product which is present in the feed: $D_{\text{lim}} = F\, X_{F,\text{lk}}/X_{D,\text{lk}}$, where the subscript lk refers to the light key component.

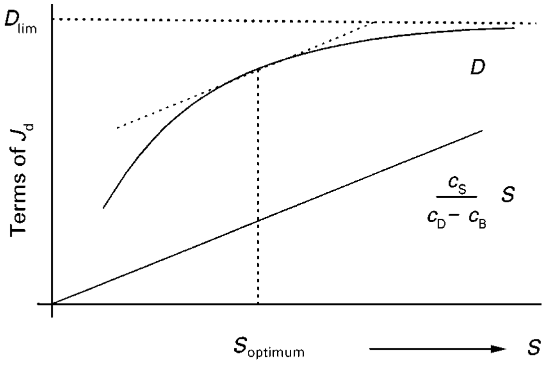

Figure 3 Optimal steam usage in distillation process.

The maximum value of J_d is found when the tangent to the curve for D parallels the straight line for $c_S S/(c_D - c_S)$, as shown in **Figure 3**. The optimum point may be located within the operation area. But at different values of the feed, however, some constraint may make the optimum point unreachable, in which case the optimum lies on the constraint.

Control of Vapour Inventory

There are a number of ways to control the vapour inventory or column pressure. Even though one of the five flows indicated in **Figure 2** could be used for pressure control, only the use of W and S gives sufficient power of control. If S were used for pressure control, a step increase in S would result (via higher pressure and top temperature) in an increase in the vapour flow in the top. Consequently, the concentration of the less volatile components in the top would increase, resulting a higher top temperature and consequently a higher vapour flow in the top. This positive feedback can sometimes be so strong that the pressure finally attains a lower value (inverse response). Therefore, the most common method of pressure control is through manipulation of the coolant flow, W.

If a water-cooled condenser is used, the water flow rate is manipulated to control pressure: if an air-cooled condenser is used, the fan speed is generally changed. The attractiveness of this pressure control option is that the condensed liquid is at its bubble point and is not subcooled, as may be the case with other pressure control options.

If there are incondensable gases in the system, pressure could also be controlled through manipulation of a bleed valve, through which the gases are bled from the column. The bleed valve is often installed on top of the reflux drum.

It should be emphasized that the pressure has a large degree of self-regulation. If the pressure in a column increases, the temperature will also increase and because of this the heat transfer in the condenser will increase; consequently the pressure will decrease again to a certain extent.

The general recommendation for pressure control is:

1. if the condenser performs partial condensation, control the column pressure by manipulating the vapour flow leaving the column;
2. if there is no net vapour flow from the column, the next preferred option is to control pressure by manipulating condenser duty, e.g. by changing the coolant flow.

Control of Liquid Inventory

Liquid inventory of the distillation process can be controlled by controlling the liquid levels: the base level in the column, h_B, and the level in the reflux drum, h_R. Often the levels serve the purpose of smoothing disturbances, hence for control of the reflux drum level the combination (h_R, D) or (h_R, R) is suitable, since W is already used for pressure control and B and S provide insufficient power of control.

For bottom-level control, D is unsuitable since the power of control is nil. Hence both (h_B, S) and (h_B, B) are suitable combinations, since they both have a large power of control.

Under normal circumstances the most logical combinations are to use the distillate flow for controlling the reflux drum level and the bottom flow for controlling the column base level.

In columns with a small distillate flow, D, and a large reflux flow, R, this scheme does not work so well. This problem can be partly eliminated by establishing a ratio controller between distillate and reflux flow. When the distillate is then increased, the ratio controller will increase the reflux flow accordingly. However, in many cases with a small distillate flow, the reflux is used for reflux drum-level control. A similar situation holds for the bottom of the column. If the bottom draw-off B is very small, level control using the liquid draw-off might not work well. In that case one could use the steam flow for base-level control. However, increasing the steam flow to the reboiler (and accompanying larger vapour flow in the column) might temporarily increase the bottom level in the column. In the long term, however, increased heat input will result in increased evaporation and consequently a lower bottom level (**Figure 4**). This response is called inverse response or nonminimum phase response and is not desirable for control purposes.

For tray columns this effect can easily be quantified using detailed column models. Assuming that any increase in vapour flow will propagate through the

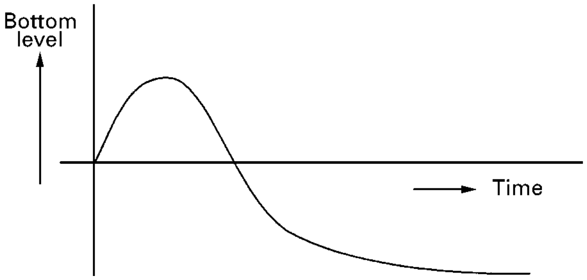

Figure 4 Bottom-level response to a positive change in steam flow.

column relatively rapidly, linearizing liquid dynamics and using deviation variables, the liquid flow from tray 1 can be written as (**Figure 5**):

$$\delta L_i = \lambda \delta V + \frac{1}{\tau_L}\delta M_i \qquad [3]$$

in which:

$$\lambda = \left(\frac{\partial L}{\partial V}\right)_{M_i}, \quad \tau_L = \left(\frac{\partial M}{\partial L}\right)_V \qquad [4]$$

where τ_L is the tray hydraulic time constant. Neighbouring trays will have similar parameter values, hence the term $\lambda \delta V$ is the same everywhere, and therefore initially all changes in liquid flows will remain the same. The mass balance on each tray will therefore initially remain at equilibrium, resulting in no change of liquid content on the trays. However, for the top tray, N, where the same flow of liquid (reflux) is still entering, a mass balance combined with eqn [4] gives:

$$\delta M_N = \frac{\tau_l \lambda \delta V}{1 + s\tau_L} \qquad [5]$$

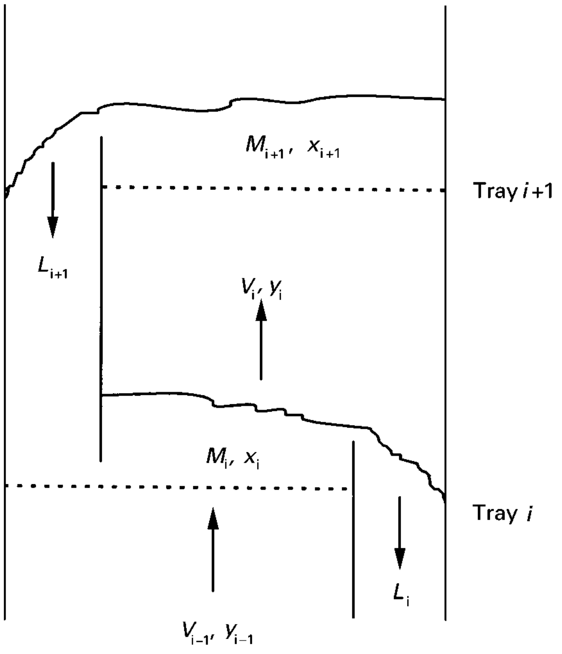

Figure 5 Schematic of a stage of a distillation column.

Substituting eqn [5] into eqn [3] and writing the result for the entire column yields for the liquid flow change for the bottom tray:

$$\delta L_1 = \lambda[1 - G_d(s)]\delta V \qquad [6]$$

with:

$$G_d(s) = \frac{1}{(1 + \tau_L s)^N} \qquad [7]$$

For the column bottom level it can then be written that:

$$\delta h_B = \frac{K_B}{s}\{\lambda[1 - G_d(s)] - 1\}\delta V \qquad [8]$$

where K_B is the gain of the bottom level in response to vapour flow changes.

From eqn [8] it can be seen that an inverse response can exist, as long as λ is not equal to zero. The magnitude of the inverse response depends on the magnitude of λ. For some columns negative values of λ have been found; for others λ is positive. It can be shown that bottom-level control on the steam (vapour flow) is strongly delayed by the effect of λ when $\lambda > 0.5$. The physical interpretation of the so-called λ-effect is that with large tray loads, an increase in vapour flow will lead to stagnation of the liquid flow and consequently λ will be larger than 1. With small tray loads an increase in vapour flow will push more liquid off the tray, thus a larger liquid flow to the lower bottom will result. **Figure 6** summarizes all options for control.

Quality Control

The response of the key components to variations in liquid and vapour flow can be approximated by the algebraic sum of the relative flow variations followed by a first-order response with a large time constant:

$$\delta x_i = \left(\frac{\delta L_{i+1}}{\overline{L}_{i+1}} - \frac{\delta V_{i-1}}{\overline{V}_{i-1}}\right)\frac{K_x}{1 + \tau_x s} \qquad [9]$$

	Basic control				
	P	h_B	h_R	x_D	x_B
W	++		+	+	+ unless $\hat{\lambda}$ >0.5
B		++ unless V_B/B>>1			
D			++ unless R/D>>1	+	
R			+	++	
S	+	+ unless λ >0.5		+	++ unless $\hat{\lambda}$ >0.5

Figure 6 Possibilities for the control scheme.

where L and V are the liquid and vapour flow respectively (see Figure 5), K_x is the gain for concentration responses and τ_x is a large time constant for concentration responses, usually in the order of hours. This time constant is approximately proportional to the square of the number of trays.

For control of the bottom quality, the reflux R or distillate flow D are unsuitable candidates, since there is a large dead time in the dynamic response between the flow and the composition. The steam flow S and coolant flow W are acceptable candidates for bottom quality control, unless, for the same reason as holds for bottom-level control, the parameter:

$$\hat{\lambda} = \frac{V_i}{L_i}\left(\frac{\partial L}{\partial V}\right)_{M_i} \qquad [10]$$

is larger than 0.5. The bottom flow B does not have a direct impact on the bottom quality; it could have some impact via a level controller.

For top quality, R, W and S are all suitable candidates for control. If, however, pressure is controlled by manipulating coolant W, the only viable option for bottom composition control is the steam flow S, after which the only remaining option for control of the top quality is the reflux R. Not in all cases, however, is there a dual composition requirement, which leaves more options for composition control.

Location of Sensors

It is important to provide the column with adequate sensors: flow measurements and pressure measurements at various locations, for example above the top tray, at the feed tray and below the bottom tray in order to be able to calculate pressure differentials for the purpose of detecting flooding. Temperature sensors should also be positioned at various locations along the column. In many cases temperatures can be used to infer composition. When the temperature difference between top and bottom of the column is small, inferring composition from temperature measurements is generally not feasible, even though in some cases temperature differences may still provide a reasonable composition estimate. An advantage of using temperature differences is that it is insensitive to pressure changes; unfortunately, the correlation between temperature difference and product composition is often highly nonlinear. In multicomponent mixtures, the relation of tray temperature to key component composition is not unique. Furthermore, the tray temperature to key product composition may also be nonlinear. Therefore, care must be taken in using temperatures for controlling composition. For high purity distillation columns sometimes the logarithm of the temperature (or composition) is used to linearize the response of the distillation column.

When the temperature difference between column top and bottom is large, several temperatures should be measured at trays above and below the feed tray, where under normal circumstances the temperature break is located. These temperatures should then be averaged and could be used in manipulating, for example, the steam flow.

An excellent treatment of sensor and valve issues in distillation control is given by Luyben (see Further Reading); the location of temperature sensors receives an especially comprehensive treatment.

For a distillation column first the base control scheme should be established, i.e. pressure, reflux drum level and column bottoms level should be controlled. Then two variables remain available for control of composition, say the reflux R and steam flow S.

If temperature is used for control of composition, the problem of proper temperature sensor location becomes prime importance.

If only one composition is controlled, a simple procedure could be followed. By giving a small change in the reflux R, the sensitivity $\delta T_i/\delta R$ can be determined for each tray. A similar procedure can be followed for the steam flow S. A typical plot for a toluene-*o*-xylene column with 30 trays is shown in **Figure 7**. It can be seen that a 1% change in steam

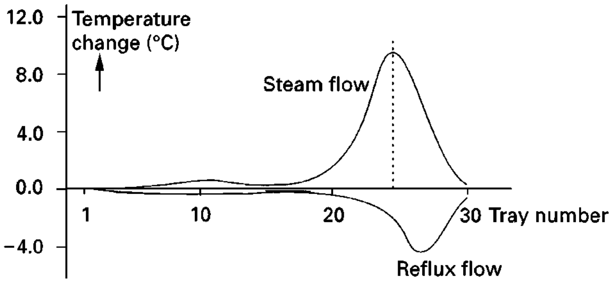

Figure 7 Gain matrix graph for the toluene-*o*-xylene column.

flow causes a temperature change of about 10° at tray 24. A 1% change in the reflux flow causes a maximum temperature change on tray 26 of about − 4°. Therefore the sensitivity of the temperature to changes in steam flow is larger than the sensitivity for changes in reflux flow. Where there is dual composition control, sensor sensitivity should be balanced against sensor interaction. One tool that has been used to accomplish this is singular value decomposition. The sensitivity matrix *X*, which contains the sensitivity of the temperature on each tray for changes in reflux and steam and thus contains two columns and 30 rows, is now decomposed into three individual matrices:

$$[U, S, V] = SVD(X) = USV^{T} \qquad [11]$$

Luyben gives a comprehensive treatment of the use of the individual matrices and their physical meaning and suggests computing a new function (combination of the principal components) for each tray:

$$Z_i = |U_{1,i}| - |U_{2,i}| \qquad [12]$$

The maximum of Z_i is an indication for the best location of the first sensor, while the minimum of Z_i gives the best location for placement of the second sensor. For the toluene-*o*-xylene column this was calculated and the results are shown in **Figure 8**. The best location for the first temperature sensor is tray 25, and the best location for the second sensor is tray 28. Note that the location has changed somewhat compared to the best locations for the single composition control problem (trays 24 and 26). This is a result of the process of reducing the interaction between the vector components at the other trays.

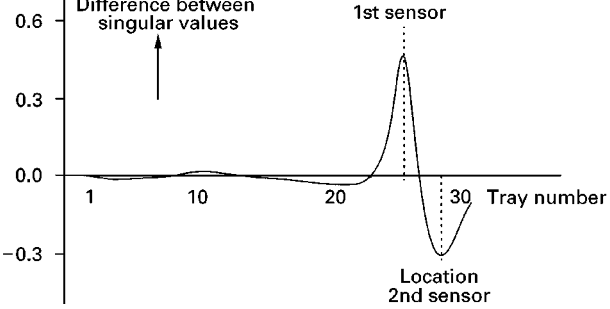

Figure 8 Sensor placement based on U-vectors.

In many columns temperature has been used to control the separation process. For binary systems at constant pressure there is a unique relationship between temperature and composition. For multicomponent mixtures, often a simple relationship may be found between temperature and composition. If the column pressure is not controlled tightly, temperature measurement should compensate for pressure variations:

$$T_{\text{compensated}} = T_{\text{measured}} + S(P_{\text{reference}} - P_{\text{measured}}) \qquad [13]$$

where *S* is the inverse of the slope of the vapour pressure–temperature curve at normal operating conditions.

No matter how attractive it is to control temperature rather than composition, the ultimate objective of the separation process is to control composition(s). This means that an analyser should be used to indicate the true compositions. There are, however, a number of disadvantages using analysers in control. First of all, analysers are highly sophisticated instruments and are therefore expensive and require extensive maintenance. In addition, the sampling system of analysers is prone to malfunctioning and as analysers are often used for multiple streams the response can exhibit a large dead time. This means that simple feedback control using analysers often results in poor control performance and dead time compensation techniques often have to be used to improve performance.

Analysers are well suited for use in a cascade control set-up, where the temperature (or combination of temperatures) controls one of the flows and the analyser controller resets the temperature controller setpoint. **Figure 9** shows one possibility, using the reflux as manipulated variable.

Control Configurations

From **Figure 6** it is clear that controlling the top composition by the reflux (*R*) and the bottom composition by the vapour flow *V* (or steam flow *S*) is just one possible option. This control option is called the *RV* configuration or energy balance structure and it is probably one of the more frequently used options for dual composition control in many distillation columns. In the literature other options for controlling

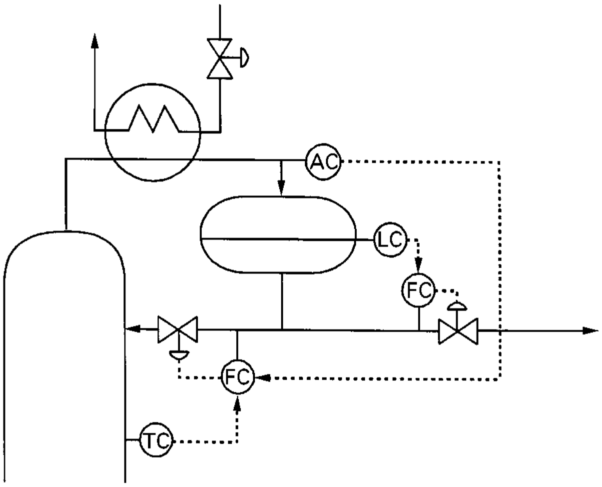

Figure 9 Composition control in a cascade structure.

both top and bottom composition are discussed and compared, such as the *DV* configuration and *RB* configuration, which are called the material balance structures.

Use of Feedforward and Decouplers

In many cases the top and bottom composition control loops will show interaction and it is advisable to design and implement decouplers. Depending on the required purity of the products and on the selected control configuration, decoupling is sometimes difficult to achieve. However, for many industrial distillation columns, decoupling considerably improves the performance of the composition control loops **Figure 10**.

The main source of disturbances usually enters the column with the feed. Whereas the feed composition may vary somewhat, the feed flow varies considerably in many cases. In those situations it might be worthwhile applying feedforward control. If the reflux is controlling the top composition and the steam flow the bottom composition, then feedforward affects both these flows on a column feed change. The principle for the reflux controller is shown in **Figure 11**, which shows a feedforward controller for feed flow changes. In a similar manner, a feedforward controller for feed composition changes could be implemented. Reflux affects the tray temperature, say via a model G_R. If the feed affects the tray temperature via a model G_F, then the feedforward controller has the structure G_F/G_R. One word of caution is necessary: care should be taken to identify properly both models, since a poorly designed feedforward controller may cause poorer control performance than no feedforward controller at all.

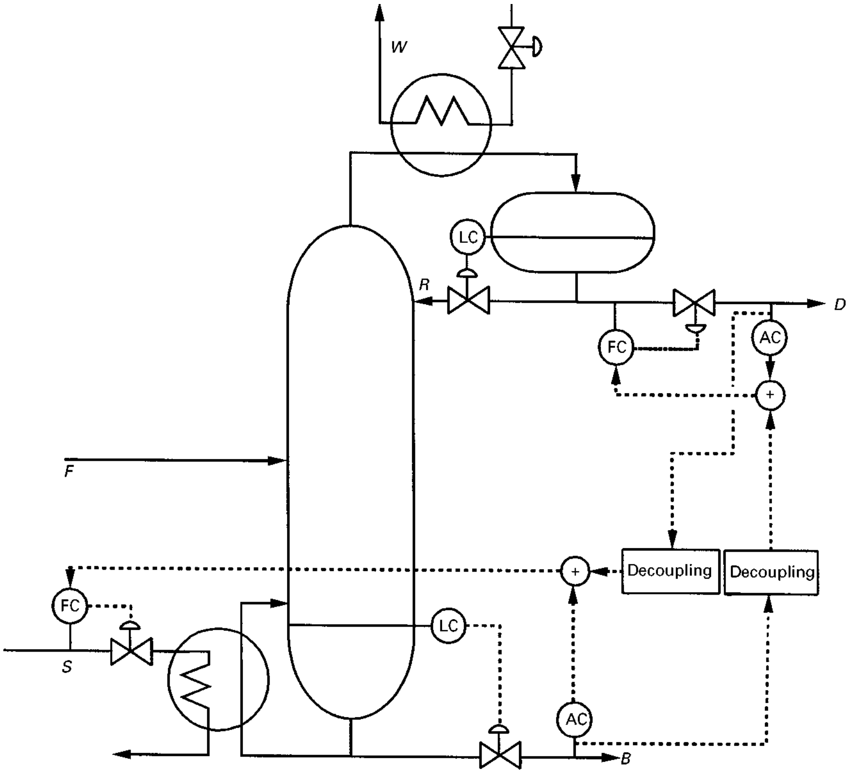

Figure 10 Example of dual analyser control using *DV* configuration with decoupling.

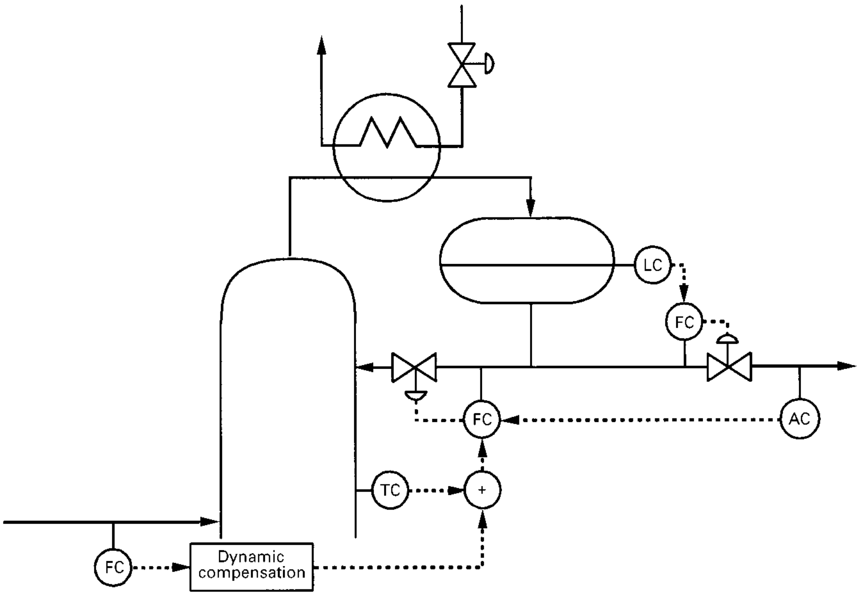

Figure 11 Principle of feedforward control.

Ratio Control

From eqn [9] it follows that keeping L/V constant will reduce variations in the concentration. **Figure 12** shows this ratio control implemented. Usually one cannot directly measure the vapour flow V, but a reasonable estimate can be made in many ways, for example from a static heat balance over the condensor:

$$V_{\mathrm{top}} = F_{\mathrm{water}}\, c_{\mathrm{water}}(T_{\mathrm{water,in}} - T_{\mathrm{water,out}})/\Delta H_{\mathrm{c}} \quad [14]$$

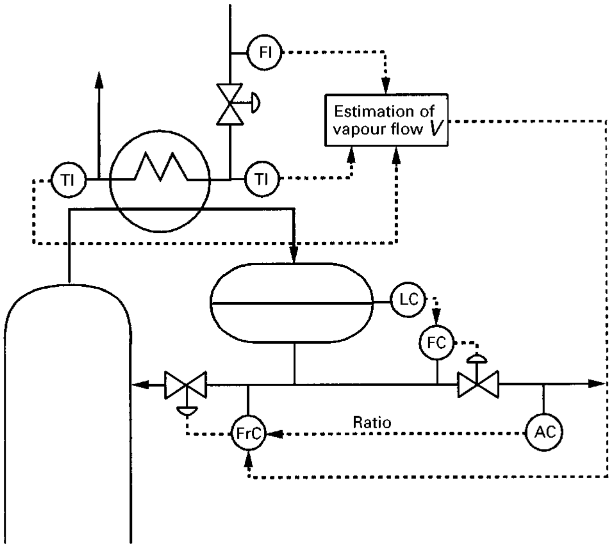

Figure 12 Ratio control between vapour flow and reflux flow.

in which ΔH_{c} is the heat of condensation and c_{water} is the specific heat of water.

Although this estimate of the vapour flow is not dynamically correct, the quality controller can be tuned such that the control scheme works well.

Multivariable Control

It is possible to consider the 5×5 control problem as an integrated problem for which an integrated controller should be designed. One technique which tries to accomplish this is multivariable predictive control. In this design all the input–output relationships of the process are identified by means of proper plant testing. Based on the models, a controller is designed, which adjusts all five process inputs simultaneously utilizing all measured process outputs. In its unconstrained version the controller is:

$$\Delta u = (A\Gamma A^{\mathrm{T}} + \Gamma_{\mathrm{m}})^{-1} A^{\mathrm{T}}\Gamma e \quad [15]$$

in which Δu is a vector with the changes in process inputs, A is a matrix with step weight coefficients which represent the model dynamics, Γ is a diagonal matrix with weights representing the relative importance of the process outputs, Γ_{m} is a diagonal matrix with penalties for the process input changes and e is a vector of process output error predictions into the future.

Model predictive control works well for distillation columns; it becomes more attractive for larger systems, such as heat-integrated distillation columns.

One of the major benefits of model predictive control is its capability in handling constraints. In that case the control problem is usually solved using quadratic programming or some other optimization technique. Commercial software packages are available for model identification and controller implementation.

Conclusion

Instrumentation and sensor location has been discussed for a distillation column. In addition, a comprehensive treatment is given of the various options for control. It is shown that some understanding of column dynamics is necessary in order to select the proper control schemes.

See also: **I / Distillation. II / Distillation:** Historical Development; Theory of Distillation.

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Laboratory Scale Distillation

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Distillation on a scale ranging from research quantities as small as ten milligrams to multikilogram lots is commonly encountered in the laboratory. Based upon the physical and chemical properties of the substance to be isolated, in addition to those of the attendant impurities and the quantity of impure product to be distilled, a technique can often be chosen which will result in a product of adequate purity in one operation. It may be said that planning a synthesis should include consideration of the difficulty one may encounter in separation of the mixture of products resulting therefrom; on the bench several alternate routes to the desired product may be available, and the resultant mixtures will differ in ease of separation. Since the quantities are not large, more expensive reagents may be chosen, if desirable, than would be acceptable in a manufacturing process. A major saving in time and effort can often be thus effected.

Resort is commonly had to three broad classes of distillation, steam distillation, flash or simple distillation, and fractional distillation. The first, finding use in separation of substances volatile with steam from those which are not steam-volatile, often uses the basic equipment of simple distillation described below, with steam being sparged into the distilland; either the product is collected as part of the condensate of the residual distilland is enriched in the desired product by removal of steam-volatile impurities. This technique, when applicable can be a powerful and convenient method requiring less skill and attention than fractional distillation.

Simple, or flash, distillation is in general used to separate individual compounds from mixtures consisting of substances whose boiling points differ by at least 40°C, and mixtures of volatile and nonvolatile components. The method consists of simply boiling the mixture in a vessel equipped with a device, commonly referred to as a 'head' which conducts the vapour to a condenser wherefrom the resultant liquid is collected. The head is usually equipped with a means by which the vapour temperature may be