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.

Further Reading

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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 observed. No forced reflux is used in this method of rectification; all the vapour reaching the head is condensed as efflux. In order to minimize entrainment of droplets, some form of dephlegmator can be interposed between the pot and the head. This technique can be conducted at pressures ranging from atmospheric to the lowest pressure available; pressures as low as 0.01 torr are not unusual. Very small quantities of product can be distilled using a device known as a 'kugelrohr (ball-tube) apparatus'; it is constructed of two or more spherical globes connected linearly by glass tubing to each other and to a vacuum source. The globe most remote from the vacuum source is charged with the distilland and vacuum is applied; the unit is rotated by hand in a horizontal position while the globe containing the distilland is heated by flame or oil bath. The vapour condenses in the globe nearer the vacuum source.

It must be stated that the development of preparative gas chromatography, a powerful and convenient method, has in most cases obviated the necessity of distillation of quantities less than 25 g. Separations hardly realizable with the most sophisticated and cumbersome techniques can be accomplished in minutes using this method; the fractions taken are usually of a high degree of purity as isolated.

The remaining method, fractional distillation, is normally used to purify quantities of crude product larger than approximately 100 g, although apparatus such as the Podbielniak column, constructed of a metal helix fitted snugly into a small-diameter glass column, have been utilized successfully to fractionate amounts less than a tenth as large. The seperation efficiency of such a column can be very high but throughput is very low. As stated, preparative gas chromatography is currently the method of choice for small quantities. For rectification of larger quantities a variety of apparatus is available, and the choice of equipment rests upon the predicted difficulty of the separation to be accomplished. As mentioned, on the bench one may be able to choose a synthetic scheme which results is an easily purified crude product; when this is not feasible recourse is had to a more efficient system capable of separating components whose boiling points lie close together.

As a rule of thumb, if the components of a mixture differ in boiling point by at least 30°, satisfactory partition can be achieved by use of Vigreux column, which is constructed of a glass tube 16–25 mm in diameter 20 cm to 1 m in length having tiers of indentations spaced 1.5–2 cm apart. The fractionating efficiency of the Vigreux column is quite good considering the ease and economy of construction of the unit.

Due to its relative openness the throughput of such a column is quite high, and as a result this type of column has become the workhorse in many preparative laboratories; its efficiency may be further enchanced by operating it under forced reflux. Throughput of 0.1–1.5 L h^{-1} are common when using this column. An additional advantage to use of this column is the ease of repair; most repairs can be made in the laboratory.

In cases where the boiling point differentials between component is less than 30°C, use of a more efficient column to effect partition will usually be found advantageous. These units are of three basic types; the first in which contact between liquid and vapour in the column is forced by mechanical construction of the column, the second in which the structured packing is used to present a large surface area to promote vapour–liquid interaction in the column (such as a bubble-cap column), and the third, a column filled with glass or metal objects of various geometries such as metal saddles, glass beads, and glass helices. The last type is often referred to as 'dumped packing'.

Units of the first type, whereby the vapour and liquid are forced into contact by the construction of the column are fairly efficient and can be designed for a reasonably high throughput. They are, however, very expensive and quite fragile when constructed of glass; thus, while they have found major application in industrial processes, where the construction is only of metal, in the laboratory they have not found use to the extent to which packed columns are used. Repairs to glass columns of this type are difficult and expensive.

The second type, structured column packings, are usually made up of a fairly closely woven metal mesh, which is then crimped and rolled into cylindrical sections which are pushed into a glass or metal column; the packed length of such a column typically will be 0.6–3.0 m for laboratory use, with diameters of 25–100 mm. The packing itself can be fabricated from many different metals, for instance stainless steels, monel, or tantalum depending on the chemical nature of the product to be purified. The advantages of this type of column are relatively high throughput, low pressure drop and moderate to high fractionation efficiency. They can be designed to operate at pressures as low as 0.1 mmHg and even greater than atmospheric pressure. In practice, however, the high vapour velocities and low vapour densities encountered at pressures less than 1 mmHg result in significant degradation of column efficiency, especially if a moderate throughput is required. As to size, a column of this type whose internal diameter is about 25 mm would be suitable for use with a distillation pot of 5 L capacity, while for use on a 50 L pot a column of 75–100 mm internal diameter would suffice. In addition, this type of column is quite easily constructed, easily repaired if broken, and can be used to predict the performance of an industrial unit of similar construction. In the laboratory, throughput of 0.1–2.0 L h^{-1}, depending on pressure, can be expected.

The last group of columns, the so-called 'dump packed' type, are as a group the most efficient at fractionation and can be the most tedious to use. They can be filled with a variety of packings, from Raschig rings (short sections of glass tubing) to columns packed with single turn glass helices dropped individually into the column. The latter is one of the most efficient fractionating columns ever devised, but as will be seen, is more suitable for distillation of smaller batches. In practice, columns of this design intended for laboratory use are 0.6–2.5 m in length, with internal diameters of 15–60 mm. Packing fabricated of perforated metal should be purchased after consultation with the manufacturer; a nominal size of 0.4–1.0 cm is usually chosen for bench use. Of the various packing components, a column filled with glass helices of 4.5–8.0 mm diameter has proven to have the greatest efficiency per unit column length but this advantage is offset to some extent by the low throughput and high pressure drops attendant with use of these units. Raschig rings are cut to a length approximating their external diameter from glass tubing 6–12 mm in size. Columns filled with dumped packing have one advantage other than efficiency in that they can easily be emptied and refilled with some other type of packing should such be desirable; conversely, structured packing, once installed can be difficult if not impossible to remove from the column without out ruining it. The disadvantages of dumped packings are a tendency to form channels through which vapour can pass without contacting liquid, low throughput, and, in most cases, high pressure drop, in addition to being prone to flooding. As a result these columns are seldom used at pressures below 10 mmHg; fractionation efficiency and throughput suffer markedly at lower pressures. In spite of these disadvantages columns of this construction have found use in many laboratories because of their ability to successfully perform separations not possible with columns of other design. Throughputs of 0.025–0.5 L h^{-1} can be expected from these columns.

General Comments

The efficiency of any distillation column is dependent to some extent upon its being operated under adiabatic conditions. Thus, distillation columns are usually insulated, enclosed within a silvered vacuum jacket, or enclosed within a heated jacket the temperature of which can be measured and controlled to within about 3°C of the vapour in the column. Too much heat will result in superheating of the vapour, causing insufficient condensation and reboiling as the vapours proceed up the column; too little heat can result in excessive condensation and flooding in the column.

Since maximum efficiency also depends upon operation at or near thermal equilibrium it is important to be able to change from one fraction to another without interrupting the distillation. A number of designs are available from scientific laboratory glass supply houses to accomplish this. In this connection, it will be found convenient to install some form of manostatic device in the vacuum line to prevent pressure fluctuations during the course of the distillation.

The source of heat input to the pot must be provided with various zones such that the heat input to the flask may be restricted to the area of the flask immersed in the distilland as the distillation proceeds. Failure to control the heat input in this way will result in serious superheating of the vapour as the distillation nears its end; driving superheated vapour into the column will result in significant loss of efficiency.

Finally, allusion was made to use of forced reflux during fractionation; it is exceedingly important to the maintenance of column equilibrium and thus fractionation efficiency. Even with a simple column such as the Vigreux, use of a partial takeoff head will result in increased ability of the system to furnish fractions of relatively high purity. In the case of packed columns control of reflux is absolutely essential to proper column performance; in its absence vapour-liquid equilibrium is never established resulting in loss of up to 90% of the fractionating efficiency of the column. Typically, reflux ratios (reflux ratio is defined as the ratio of the amount of condensate returned to the column to the amount of condensate collected as efflux) as high as 50 : 1 are not uncommon during difficult separations; ratios approaching 1 : 3 are sometimes used during centre cuts or end fractions. Various means to effect reflux control are available from laboratory glass suppliers, from simple devices utilizing manual control by means of a stopcock to elaborate units in which a magnetically controlled value is used to divert the condensate stream either to the column or to the receiver.

Caution

Since a difficult fractionation may consume days, during which the pot is at constant reflux, heed must be paid to considerations of thermal stability in the distilland, especially since small quantities of acids or bases can, and do accelerate thermal degradation of some organic chemicals. In fact, some thermal decompositions have been shown to be autocatalytic. It therefore may be well to consider flash distillation of the crude product prior to subjecting it to fractional distillation to remove trace non-volatiles and/or non-volatiles which would accelerate decomposition or lead to excessively high pot temperatures. In some cases one might consider the addition of a stabilizing agent to the pot to retard decomposition.

Closing Remarks

With all of the above having been stated, fractional distillation, particularly at reduced pressure, can be viewed as an opportunity to see physical chemistry at work. When selecting a system one hopes will result in satisfactory partition of components it will be helpful to consider properties other than the boiling point. For example, if a mixture of intermolecularly bound substances is to be separated by distillation, their partition is likely to be more difficult than the differentials between their boiling points would indicate. On the other hand, a mixture of alkanes may well be more easily separable than comparison of their boiling points would otherwise indicate. In any case practice is necessary, both conducting distillations and selecting systems for distillation. Once experience has been gained it is satisfying to be able to rationalize the results of a fractionation in terms of physico-chemical principles. One positive note: since distillation does not result in loss of product, in the worst case one can recombine all the fractions and redistill using different conditions and, if necessary, a different system.

Modelling and Simulation

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Introduction

Rigorous computer modelling of all types of fractionation columns has become a necessary part of the development and design process. There are numerous software products available to do these calculations. An understanding of the basic mathematics used in these programmes is helpful to select, use and troubleshoot a column model. Explained here are the basic equations, numerical and solution methods commonly used.

Stage and Column Models

A rigorous method describes a column as a group of equations and is the mathematical engine to solve and satisfy these equations to calculate the operating conditions of the column.

Column design and performance calculations present the column at steady state, that is, what enters the column matches what leaves it (material and energy balances), i.e.:

$$\sum(\text{molar feed flow rates}) = \sum(\text{molar product flow rates})$$

$$\sum(\text{mass feed flow rates}) = \sum(\text{mass product flow rates})$$

$$\sum(\text{moles of any component in the feeds}) = \sum(\text{moles of the component in the products})$$

$$\text{Feed enthalpy} + \text{Heat added} = \text{Product enthalpy} + \text{Heat removed}$$

Figure 1 shows a complex column with one feed and one side product. The top stage of the column is a partial condenser, with a vapour product, D, and a liquid product, d. The reflux is the liquid, L_0, and the reflux ratio is $L_0/(D + d)$. The bottoms product, B, leaves stage N + 1, the reboiler. The stages are numbered from the top, with the condenser as stage 0, the top tray in the column, stage 1, the bottom tray, stage N, and the reboiler, as stage N + 1.

An ideal or equilibrium stage is where vapour and liquid entering and leaving the stage are perfectly mixed and there are no inhibitions to material transfer between the phases. The material and energy flows in and out of a simple stage, with no feeds or side products, is stage j depicted in Figure 2, and i represents the component number. Components are numbered from 1 to the last, C.

The enthalpy terms, H_j and h_j, are molar enthalpies of the vapour and liquid leaving the stage, respectively. These molar enthalpies are multiplied by the total flow rates, V_j and L_j, leaving the stage to give the total energy leaving the stage in each phase.

The feed stage model (stage f in **Figure 2**) for an equilibrium stage assumes that the feed liquid mixes