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# Distillation: Research Needs

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# SPECIAL REPORT

# Distillation: Research Needs

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**Abstract**

Distillation will undoubtedly continue to be the most-used method for separating liquid mixtures, at any scale of operation. For this reason, and also because of its recognized energy intensiveness, distillation commands continued scrutiny with respect to cost-effective improvements. In this special report the authors suggest fruitful areas of research that can lead to lower cost distillation separations. The areas of research are classified under the headings of phase equilibrium, material and energy balances, mass transfer efficiencies, equipment design, and system energy consumption. For each of the categories a summary is given of the present status of the technology as well as directions that improvement-type investigations might take.

## INTRODUCTION

Distillation is well known by all chemical engineers. Its fundamentals are taught in all undergraduate programs. The published literature relating to distillation is enormous. As a technique for separating fluid mixtures, it pervades the chemical and petroleum industries. It has been termed the workhorse of the commercial separations technology effort. And it represents a baseline case against which other separation methods must be measured. Thus, there is some compulsion to make a report of this type quite brief, with the general theme, "We know all we need to know about distillation, and therefore there are no needs for further research."

Closer examination, however, suggests that there are indeed some things about distillation that we do not know, and that we should know, considering the huge investment in distillation that is in place as well as the likely continuing large expenditures on distillation as a major separation



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method in new plant facilities. Further, distillation has become known as a highly energy-intensive separation method, and it would seem that any new knowledge that might lead to energy reductions in distillation design would be worthwhile to seek.

In this report no attempt is made to summarize the state-of-the-art of many aspects of distillation technology. Only those aspects thought amenable to improvement through additional research are covered in any detail. The assumption is made that the reader is familiar generally with the principles of distillation, the type of equipment used for commercial separation needs, and the general areas of application of the method. Research needs are discussed under the headings of phase equilibrium, material and energy balances, mass transfer efficiencies, equipment design, and system energy consumption.



## PHASE EQUILIBRIUM

When distillation is considered for the solution of a separation problem, the first question asked is, "Is there sufficient volatility difference between the species to be separated?" Not only is the answer to this question the key to the separation method selection, but its rigorous support is the foundation for a rather good-sized continuing research effort in the general area of phase equilibrium. For distillation, of course, the effort is in vapor–liquid equilibrium (VLE), and laboratories at the University of California at Berkeley (Professor J. M. Prausnitz), Washington University (Professor B. D. Smith), and Rensselaer Polytechnic Institute (Professor H. C. Van Ness) are typical of those throughout the world that are generating useful equilibrium data for nonideal liquid mixtures. A key activity of workers in this field is the development of predictive methods for nonideal cases, with the approximate goal of having the ability to eliminate experimental work completely and rely on predictive models for handling not only simple binary mixtures but also multicomponent, highly nonideal systems.

A major effort is being expended on the development of the UNIFAC group contribution method (*1, 2*) for VLE prediction. This research involves the experimental determination of liquid-phase activity coefficients for a number of binary pairs that contain appropriate functional groups, in order that the contribution method can be validated for many possible combinations of groupings. To this end the AIChE Design Institute for Physical Property Data (DIPPR) is coordinating the accumulation of equilibrium data, with funding at a number of university and private laboratories. Status reports on the progress of the UNIFAC group

development have been published recently (*3, 4*). Background on the DIPPR operation may be obtained from the paper by Kemp (*5*).

A certain amount of empiricism is inherent in the UNIFAC method, and it cannot be regarded as entirely reliable for new and unusual systems. If such systems involve low relative volatilities and correspondingly large numbers of equilibrium stages for a desired amount of separation, recourse to experimental data will be required. The correlation and extension of such data can be handled adequately by one of several available nonideal binary models, such as those of Wilson (*6*), Renon and Prausnitz (*7*), Van Laar (*8*), and Margules (*9*). The fit of these models to experimental data has been discussed by Null (*10*) and by Holmes and Van Winkle (*11*), among others. It is possible to build multicomponent equilibria from binary pair data (*10*), even using different models for each pair (*12*).

There has been significant effort in compiling and evaluating previously reported vapor–liquid equilibrium data. A series of bibliographic source books have been published by a group at Prague, Czechoslovakia (*13–15*). Data listings, as well as values (with correlation fits) of the parameters for the Van Laar, Magules, Wilson, and Renon-Prausnitz (nonrandom, two liquid phase, or NRTL) models, are being published in a multivolume set sponsored by the Dechema in West Germany (*16*). A less-ambitious listing project, covered in a single volume, is that of Hirata, Ohe, and Naghama (*17*).

In summary, current work is concentrated on the compilation of VLE and the development of estimating parameters for the UNIFAC predictive method. Both of these efforts are useful for many designs encountered by process chemical engineers and will result in less need for laboratory data. There is a need for this work to continue to some reasonable conclusion. Sufficient models are available for the extension of experimental values of nonideal equilibria. One would not expect new, major efforts to begin in the phase equilibrium area.

## MATERIAL AND ENERGY BALANCES

Work in the area of stagewise contacting has been directed toward the rigorous calculation of equilibrium stages for distillation columns that have one or more feeds and one or more sidestream drawoffs (Fig. 1). The models have been programmed for computer solution, and it is almost routine to handle separations involving 100 stages or more and mixtures up to 25 or more components. The solutions provide detailed information on concentration, temperature, and pressure profiles. For some cases there are

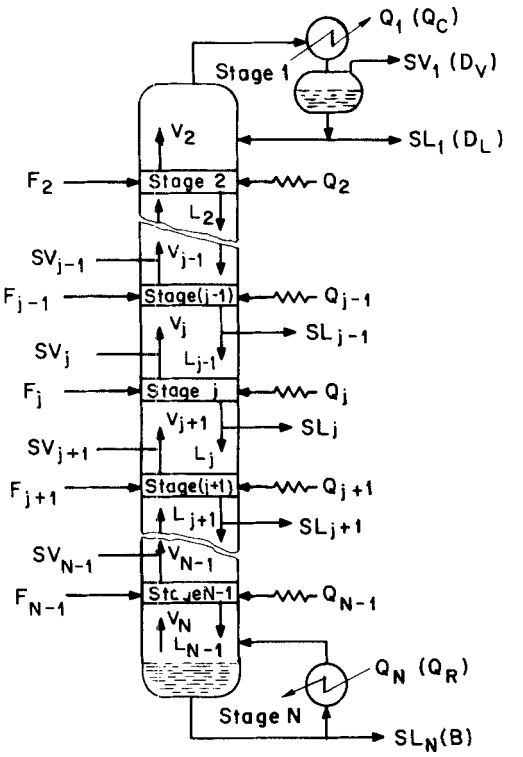


FIG. 1. Generalized multistage separation column (*19*).



convergence problems, but by and large this segment of distillation technology seems well under control. Detailed discussions of these rigorous methods are contained in the book by Holland (*18*), the paper by Wang and Wang (*19*), and other similar references. For the so-called short-cut method for determining theoretical stages, rigorous methods for obtaining minimum stages and minimum reflux have been published by Chien (*20, 21*), and the combination of these parameters to produce working plates vs working reflux relationships has been studied by Erbar and Maddox (*22*) using multiple computer solutions. The methods for minimum stages and minimum reflux are important also for rigorous calculations, since they set the limits on the plate vs reflux relationships for a given separation.

It is not clear whether any significant needs for research remain in this area. Developmental work continues in the models for stage determination (material and energy balance calculations) to improve convergence for inherently unstable systems. In addition, such models have been incorpor-

ated into full process simulators such as those of ASPEN, FLOWTRAN, PROCESS (Simulation Sciences), and DESIGN (Chemshare). Mention of these simulators reminds one also of continuing work to provide physical property data banks for direct access by the simulators. Thus, needs in the area are centered on marginal improvements of computer algorithms and on the continued expansion of physical property data bases.

## MASS TRANSFER EFFICIENCIES

For many years it has been rather a joke among process engineers that so much effort is expended on determination of theoretical stages, involving rigorous computer routines, and so little effort is expended on the problem of the magnitude of departure from equilibrium in actual distillation equipment. It is not uncommon for an engineer to spend a number of days to arrive at, say, a need for 28 theoretical stages. Then, in a matter of minutes, and with gross abandon of rigor, he chooses, say, an overall stage efficiency of 70% to give a nice, round number of 40 actual stages. One cannot sidestep this issue by saying, "actual stages are cheap." If this is the case, why all the trouble to calculate theoretical stages? In fact, cost of the stages may not even be the important aspect of the design; it may be cost of pressure drop (vacuum fractionation) or cost of reflux which equates to cost of energy.

For countercurrent distillation, usually performed in packed columns, the same philosophy prevails. The engineer may make a rigorous determination of required transfer units and then use an approximate value of the height of a transfer unit (supplied by a packing vendor in the less fundamental form of a height equivalent to a theoretical plate [HETP]) to obtain a total required height of packing. It would appear that an improved rationale for arriving at actual stages or packing height is needed.

The efficiency of a distillation contacting device depends upon three sets of parameters: transport properties of the mixture being separated, geometrical characteristics of the device, and the rates of flow of the contacting phases. Determination of efficiency is a complex problem of mass and momentum (and sometimes heat) transport. The two-phase mixture on the stage or in the packing is complex and far from well understood. Transport properties of multicomponent mixtures are poorly known and are difficult to handle mathematically. And too few data are available that can be used to check theoretical or semitheoretical models.

For staged fractionation, the crossflow contacting device (sieve tray, valve tray, bubble-cap tray) is commonly used. For such a device, three different efficiencies can be defined, as shown in Fig. 2. The local, or point,

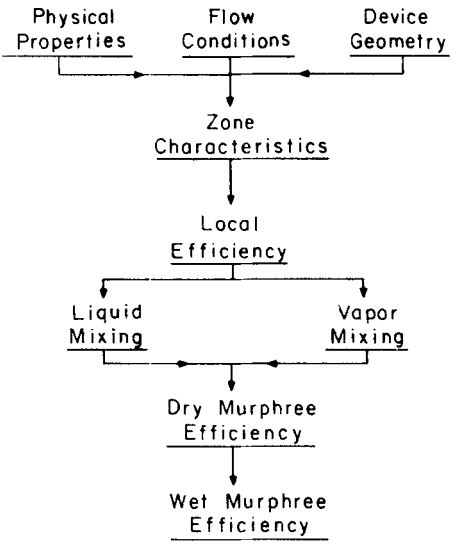


FIG. 2. Steps in the determination of Murphree tray efficiency.



efficiency is the most basic of the three and can be measured with small columns or in special laboratory devices. The other efficiencies depend upon liquid and vapor mixing conditions and on vapor flow rates that might cause entrainment of liquid to the next tray above.

It is possible to make estimates of local efficiency by means of the AIChE bubble-cap method (*23*). This model is limited to binary systems and has been shown to represent poorly the efficiency data for sieve trays (*24*). Furthermore, corrections for crossflow of liquid and its departure from plug flow must be added. Recently, an improved binary model, developed for sieve trays, was presented (*24*). This new model also predicts local efficiency, but its development also incorporated a set procedure for correcting for crossflow effects. It has also been extended to multicomponent systems (*25*), yet it is semitheoretical, and it may be limited to sieve trays.* It does not take into account, for example, the different two-phase regimes (bubble, froth, froth-spray, spray) that can exist on a contacting tray. How it handles entrainment under high vapor flow

*It is not possible here to go into the details of the various devices available for staged-type distillation columns. The sieve tray is a popular and nonproprietary device, and a state-of-the-art review of its characteristics and design has been reported recently by Zuiderweg (*36*). A description of the various devices, together with a historical sketch of equipment development, was presented recently by one of the authors (*26*).

conditions is not clear. Nevertheless, it is a great improvement over the previously available models. Clearly, our understanding of the contacting mechanisms on trays is quite poor. The question may be, "Do we need to understand the mechanisms in order to do a reasonable job of designing the equipment?"

Before attempting to answer this question, which is directed toward staged devices, it may be well to comment on the general state of research on commercial-scale distillation equipment. In the United States, such research is essentially confined to the efforts of Fractionation Research, Inc., a nonprofit closed corporation that does confidential work for a number of industrial sponsors. This organization, known simply as FRI, has in effect drawn research away from companies and from universities. A small portion of the FRI work has been published and has proved to be very useful (see, for example, Ref. *24*) to outsiders. By and large, however, the FRI data are not available and, unfortunately, FRI does not conduct fundamental mass transfer studies of the mechanistic modeling variety. In England, a well-equipped distillation test facility has been built at the University of Manchester Institute of Science and Technology (UMIST), incorporating a 24-in. diameter column. Typical test data have been reported recently (*27*) and at least one set of runs has been made on a new tray device (*28*), but apparently the cost of operating the system is such that one can expect to see very few experimental data reported.



Now, to return to the question regarding a need for understanding the mechanisms of mass transfer in distillation equipment: If one examines the details of available models, and recognizes that they are not highly reliable and that they are largely empirical, he will soon feel rather uncomfortable in using them for cases where every actual stage or height of packing is critical. The models need to be more fundamentally based, and this will require a great deal of additional experimental work. In some respects the mass transfer issue is the last major frontier in the distillation technology saga. In the opinion of the authors, it demands continued attention just as do, for example, the mechanics of falling films or the regimes of flow in cocurrent flow phase-contacting in round channels.

## EQUIPMENT DESIGN

In recent decades, distillation columns have been of two major geometries: stacks of trays or volumes of dumped (random) packings. The major tray types have been bubble-cap, sieve, and valve; all of these trays involve a crossflowing liquid contacted by an upflowing vapor. The major dumped-type packing materials have been Raschig rings, Intalox saddles,

and Pall rings. Sufficient data are available to permit determination of flow limits and pressure drop, and reasonably reliable (though mostly empirical) models are available. For the trays, models for predicting mass transfer efficiency have been discussed above. For the dumped packings, it is not uncommon for no effort to be expended on mass transfer prediction, reliance being made on the packing vendor and his estimates of HETP (as discussed earlier). However, there are three methods that utilize mass transfer theory (two-resistance approach) and form a structure upon which further studies might be based (*29–31*). As indicated, these methods are limited to conventional dumped packings such as those shown in Fig. 3.

A great deal of attention is now being given to structured-type packings that are arranged carefully before being inserted into the column. Although considerably more expensive than the dumped packings, on a volume basis their favorable efficiency/pressure drop characteristics can justify their cost for cases where pressure drop is expensive, for example, when heat-sensitive materials are being distilled under vacuum. Examples of structured packings are shown in Fig. 4. There is a great need for objective data on these devices, something not easily obtainable in a very competitive market as found today.



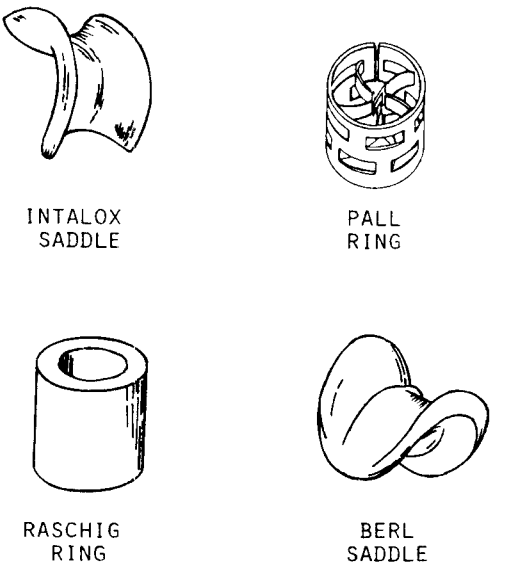


FIG. 3. Common types of dumped packings.

For the more traditional contacting devices (crossflow trays, dumped packings) there does not appear to be a pressing need for improved methods for predicting flow limits (flooding, entrainment, weeping). For trays, pressure drop models are adequate. For dumped packings, pressure drop models are poor, the only nonproprietary general method being that of Eckert (*32*); in 1979 Bolles and Fair showed that this method gave poor agreement with a special bank of commercial-scale data (*33*). Thus, the only pressing need in the traditional equipment area is for a good and general model for pressure drop prediction in dumped packings.

For the new structured packings, tests are needed in facilities such as those at FRI,* UMIST, or The University of Texas at Austin (presently being designed to include an 18-in. diameter test column). It is characteristic of these packings that they do little to improve the initial distribution of liquid at the top of the bed; thus tests must include careful studies of initial liquid distribution and the degree to which this distribution can be maintained throughout the packed bed. Earlier studies of Sulzer packing (*34*) showed that column diameter had little effect on mass transfer efficiency; it will remain for some generous commercial user, with a large-diameter bed of structured packing, to report on diameter effects beyond the test region of 40 in.

It would seem that the structured packings should be more amenable to mechanistic modeling than the dumped packings, which arrange themselves in a random fashion. Earlier work with falling films and with wetted wall columns should be useful in modeling of both the hydraulic effects and the mass transfer rates, as suggested by Hughmark (*35*). A recent presentation of pilot-scale studies of FLEXIPAC (*36*) represents a good start on the accumulation of basic data and appears to be well done and reliable even though it stems from the laboratory of the FLEXIPAC vendor. More work of this type is needed.

Some effort continues in the development of new devices that promise higher capacity, better efficiency, or lower pressure drop than devices currently on the market. In England a packed contactor that is rotated at high speed has been developed by Imperial Chemical Industries. Called the "HIGEE," it has been described generally in the journals (*37, 38*). Higher gravitation conditions permit higher throughputs, and special packings permit higher mass transfer rates. Unfortunately, there is a significant pressure drop for vapor flow through the equipment, and applications for vacuum fractionation may be limited. Little is known about capital costs,

*Sulzer packing, formed from wire mesh in a special corrugated arrangement (Fig. 4), has been tested by FRI in a 40-in. column in Switzerland. The results are available from the vendor (*34*).

but maintenance costs could be significant because equipment must be operated at high rotational speeds.

Another new device of the tray type, "Parastillation," comprises half-trays with parallel flows of liquid throughout each half-column section. Computations show that two half-trays are equivalent to about 1.3 conventional trays; this finding has been confirmed by tests at UMIST (*28*). New high-capacity dumped-type packings have been introduced, including the Norton metal Intalox (*39*) and the NSW plastic ring (*40*). There is a need for objective testing of all new devices, and FRI performs some of this service. However, the FRI results can only be released to the general public (i.e., non-FRI members) at the option of the vendor. Testing at a university, as was done for NSW packing (*40*), seems to be a better idea.

To summarize the needs for research on distillation equipment, with the recognition that current trends toward the manufacture of higher-valued



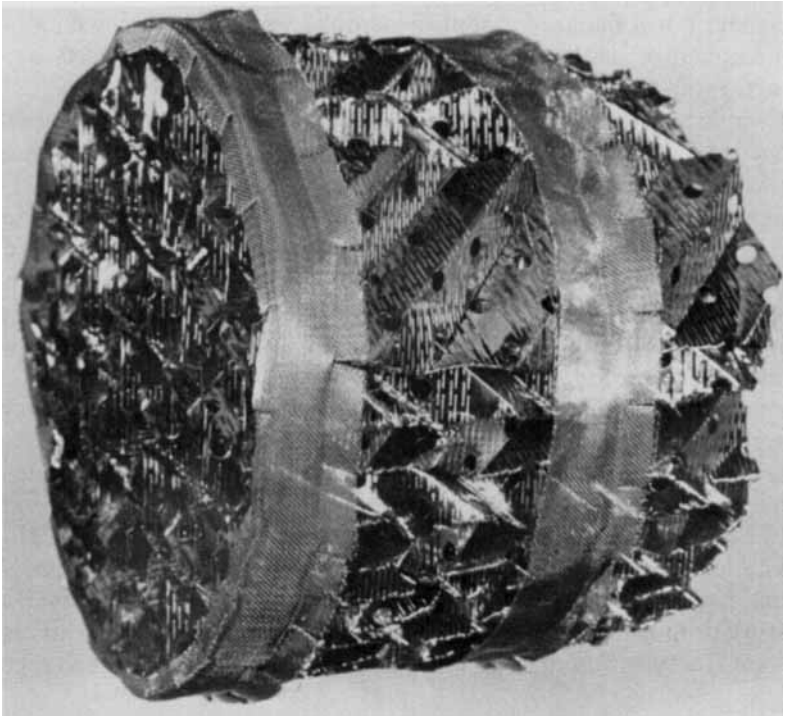

FIG. 4. Examples of structured (ordered) packings. Above: Gempak (Glitsch, Inc.). Opposite: Koch Sulzer (Koch Engineering Co.).

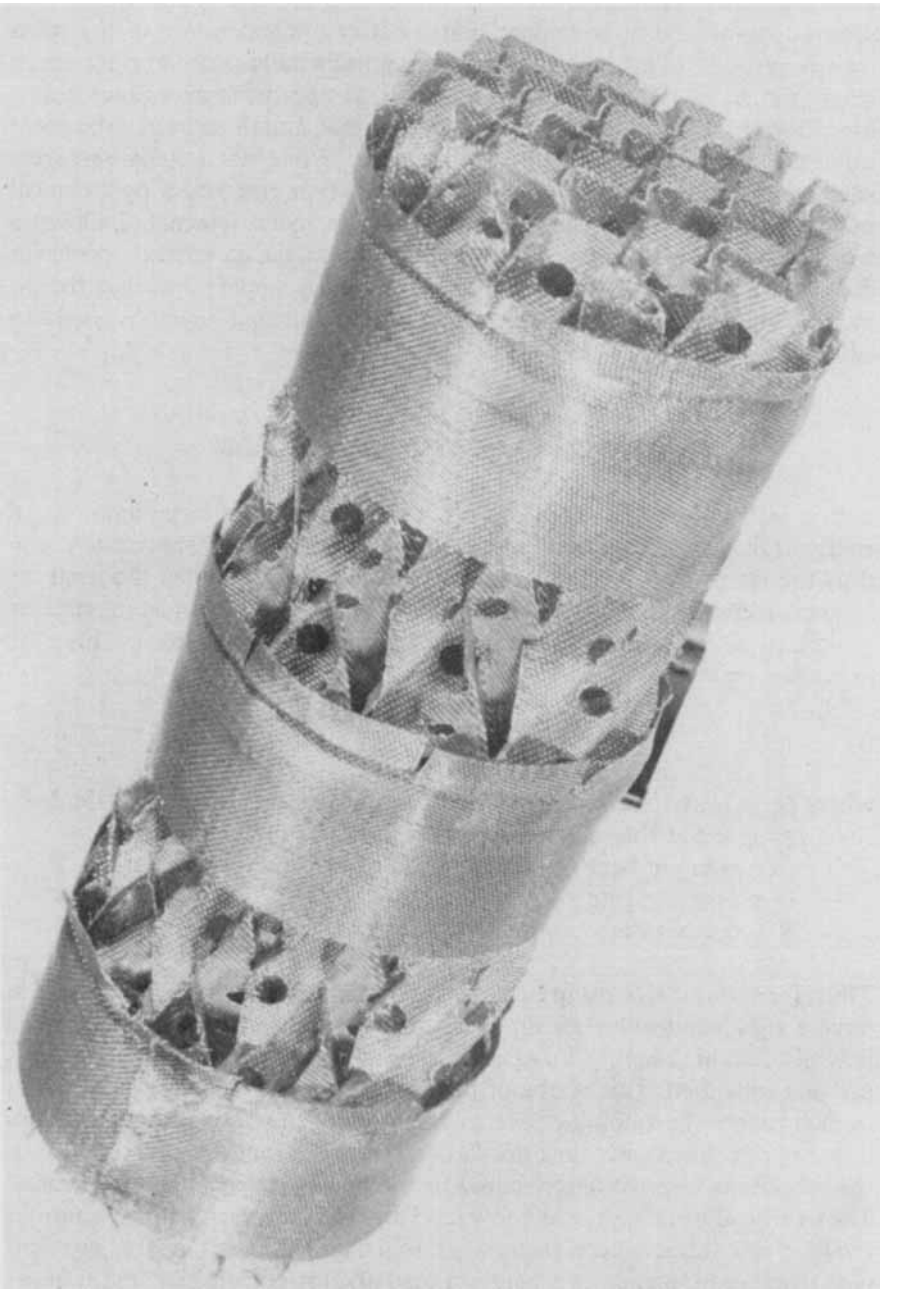

FIG. 4 (continued)

chemicals and petroleum derivatives will still require distillation as a separation method, it is evident that a better understanding of the mass transfer process in packings and on trays is still vitally necessary. Research needs should be concentrated in this area. It appears that a flow of new inventions in the way of equipment will continue, and these need to be given unbiased analysis from testing laboratories. While not strictly research, such testing should be done in good research-type equipment by technical people who appreciate the value of accurate measurements. Follow-up work on modeling would also be desirable. In fact, the contacting process in distillation columns is so complex and so poorly understood that fundamental work in the area can provide a basic challenge for many years to come.



## SYSTEM ENERGY CONSUMPTION

It is well known that distillation consumes relatively large amounts of energy. Distillation makes its separation through partial vaporization, and thus the latent heat of vaporization must be supplied. When the need for reflux is recognized, it is evident that the distillate product must in effect be vaporized several times. The energy required for this process may be expressed by

$$Q_c = D(\Delta H_c)(R + 1)$$

where $Q_c$ = heat removed at condenser, approximately equal to the heat supplied at the reboiler, feed and products in liquid state
$\Delta H_c$ = latent heat of condensation of top-stage vapor
$D$ = distillate rate
$R$ = reflux ratio

Thus, from this relationship one can see that a reduction in reflux ratio can have a significant influence on energy conservation efforts for existing or new distillation columns. Lower reflux ratios at the expense of more trays are often justified. This is one of the first axioms of the energy conservationist: reduce the amount of energy required for the distillation separation.

A basic problem with most distillation systems is that the heat supplied to the reboiler is approximately equal to the heat rejected at the condenser. The overhead heat is often at a low level and is suitable only for rejection to the heat sink (atmosphere, river, lake). In a nutshell, this is the real problem with distillation: higher level heat is added and lower level heat, in the same approximate amount, is rejected. This leads to the second axiom of the

conservationist: attempt to use the heat that is present in the overhead vapors from the top of the column.

For distillations carried out at higher temperatures, the heat in the overhead might be used to generate low-pressure steam, to vaporize a Rankine cycle working fluid, or to provide heat for another distillation column (the "coupling" or multiple-effect situation). Alternatively, the heat might be "pumped" to a higher level where it could be useful, and this leads to a discussion of mechanical vapor recompression as applied to distillation.

Mechanical vapor recompression (MVR) is not a new concept. It is not a product of the 1970s energy crisis. For many years its potential value has been recognized. Figure 5 shows a flow scheme for the technique, published by one of the authors in 1959 (*41*). A complete analysis of MVR was published in 1976 by Null (*42*).

There are several schemes by which MVR may be practiced; Fig. 5 shows only one of them. The distinguishing feature of this idea is that energy in the form of heat (from condensing steam, sensible change in a heat transfer medium, and so on) is replaced (all or in part) by compression energy. The latter may be provided by a steam-driven turbine or by an electric motor, depending upon steam availability and cost of electricity.



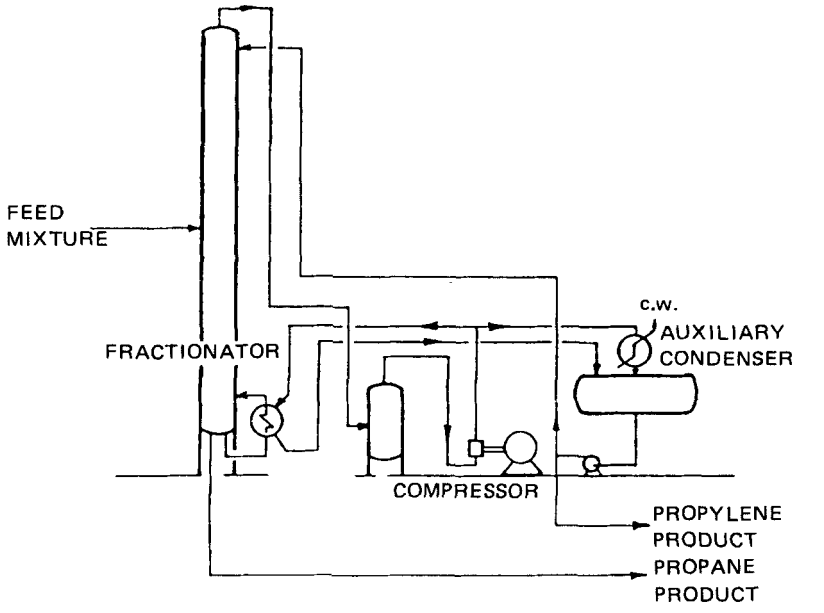


FIG. 5. Propylene and propane are conveniently separated by vapor recompression distillation at 100 to 200 psia (*41*).

It is clear that the applicability of MVR to distillation is dependent upon the economics of heating medium availability/cost versus steam/electricity availability and cost. There appears to be a need for more thorough investigation of MVR applicability. One development that could strongly influence the areas of applicability of MVR is the advent of the new high efficiency/low pressure drop structured packings, as discussed above. These packings can help bring the overhead and bottoms temperatures closer together and thus reduce the cost of pumping the heat to a level such that it can be used for the reboiler. The effect of this development on MVR economics has not been studied.

Another need in the area of MVR is that of controllability studies. There are no operating reports on commercial MVR installations, and thus there is little guidance for the process engineer who is considering the use of MVR in a new design or for a retrofit situation. Control studies need to be performed on a scale large enough to provide equipment responses that would be representative of commercial situations. These controllability studies probably should be combined with studies of coupled columns where typically one column's overhead vapor is used to provide boil-up energy for another column (*43*). The criticality of control can be envisioned for the case of a distillate product that represents a fairly wide boiling mixture in which transients have a pronounced effect on the composition of the mixture. In summary, there is a need for further investigation of MVR in an experimental mode, using relatively large equipment.



It is possible to combine other energy-conserving ideas with MVR in order to improve further the energy efficiency of a distillation column. An example of this is the secondary reflux and vaporization method of Mah and co-workers (*44*), as shown in Fig. 6. A close look at this figure shows that the rectifying section of the column operates at a higher pressure than does the stripping section. This arrangement enables thermal coupling of the two sections to provide intermediate condensers and reboilers, thus reducing the needed external reflux ratio. Energy is needed for the MVR compressor and the stripping section vapor compressor, but the total heat requirement for the separation is reduced. This is just an example of "paper studies" that need to be encouraged, even though the capital cost and operating difficulty factors may seem insurmountable.

In general, analyses may be carried out along lines suggested by King (*45*) and result in values of the minimum work of separation (free energy change) compared with the network of separation that is derived from an availability analysis. Thus, a continued need exists to explore improved configurations of distillation systems that lead to basic energy reductions. One must also recognize that existing distillation columns are often operated at higher reflux ratios than are really necessary, in order to stay

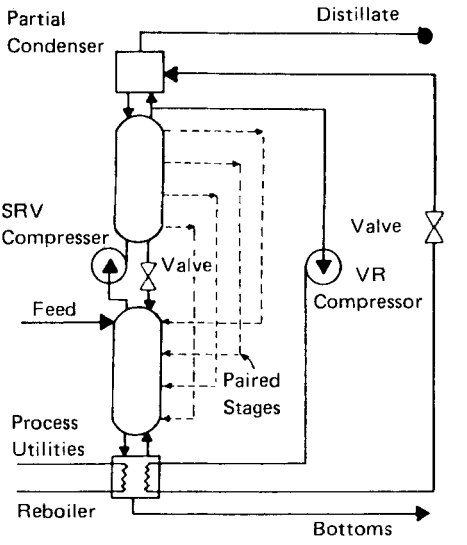


FIG. 6. Distillation with secondary reflux and vaporization (*44*).

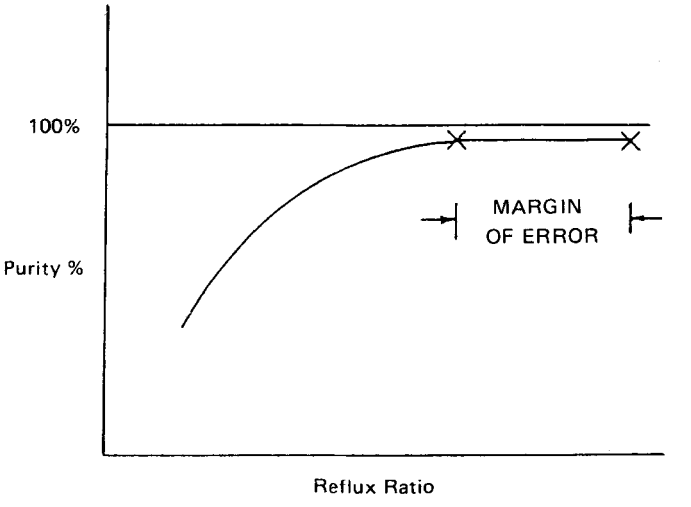


FIG. 7. Distillation columns operating in the zone noted as "margin of error" are wasteful of energy.

well within a "margin of error" as shown in Fig. 7. Careful surveillance of plant operating policies can lead to significant amounts of energy reduction.

A final thought for this section is one that cannot go unmentioned. The large majority of existing distillation columns use low-pressure steam as a heat source. The steam has, very likely, been sent through one or more users (turbine drives) before reaching the distillation reboiler. It seems almost characteristic of large chemical and refining complexes that there is a chronic surplus of low-pressure steam. In other words, it is difficult to design initially a completely balanced steam system for a plant that invariably undergoes changes in operating rates and in processing unit additions and deletions. Because of this characteristic, it is often difficult to justify spending a great deal of money on an existing column to reduce its steam consumption. In a sense, the distillation column reboiler performs a useful service in condensing surplus steam for reuse in the power plant. This same line of reasoning might not apply to new designs, but it does suggest the need for further study of steam balances in complex plants. Such study must, however, relate to the proprietary needs of the plants involved and may not be amenable to a generalizing effort.



## SUMMARY

For *phase equilibrium* studies the major needed thrust is in the further development of methods for predicting vapor–liquid equilibria with little or no need for direct experimental work. It would be desirable for such methods to be fundamental in their consideration of molecular shape, size, and interaction among dissimilar species. On an interim basis it will be necessary to make some compromise with this desire, and to this end the UNIFAC approach shows most promise at present. To support UNIFAC or another, more fundamental, method, it will be necessary to provide a continuing flow of reliable equilibrium data, taken under careful conditions and subjected to thermodynamic consistency analyses. Work in progress appears to be satisfying the need for additional data.

For *material and energy balance* studies there is some need for improved algorithms to provide better convergence of certain basically unstable computation situations. Such work is in progress, some of it in academia and some of it in connection with service bureaus offering full process simulation capabilities. There is not an equivalent effort for determining transfer units in counterflow contactor simulations, but conversion from equilibrium stages to transfer units appears to be satisfying the present need.

For *mass transfer efficiency* studies there is a great need for better understanding of the contacting mechanisms that occur on trays or in packings. There appears not to be enough research in progress to begin to satisfy this need. One deterrent is the very complex two-phase flow situation that occurs on trays and in packings, and the necessary experimental work to elucidate the fluid mechanics and mass transfer will not be accomplished easily. A complicating factor is the wide range of contacting devices that are used commercially for effecting intimate contact of vapor and liquid. It seems advisable to limit current work to crossflow sieve trays and counterflow structured packings. These devices are not only the most popular ones being considered today, but the most amenable to mechanistic modeling for predictive purposes.

For *equipment design* and development, there is a need for additional performance data, taken at a commercial scale, on the better-known and higher-performing devices. As mentioned just above, these include the sieve tray and the structured packings. Some additional data on random packings would be useful in supporting the development of methods for predicting pressure drop. Work of the type done by Fractionation Research, Inc., will be important to the future development of new contacting devices, since such work can be free of the bias provided by the developer/inventor of a new device. In general, a better understanding of the hydrodynamic and mass transfer characteristics of the various devices represents a research need in the equipment area.

For *system energy consumption* studies, there is a need for continued improvement in the energy efficiency of distillation columns, and in this context is included means for decreasing the required energy for distillation separations. Approaches to answering this need are amenable to thermodynamic analysis, and this is, of course, the first step to be taken. However, some of the ideas fall down when subjected to analyses that consider cost and practicability. Here is where some larger scale experimental work will be required. A good contender for energy reduction in distillation is mechanical vapor recompression, but there may be problems of scale-up and operability/controllability that need analysis under pilot-scale or commercial-scale conditions of experimentation. Thus, the needs in the energy area can be expressed simply: more thermodynamic analysis and more experimental testing.

Overall, it may be concluded that distillation will remain as a key method for separating fluid mixtures and will prevail even if there are major "restructuring" changes in the chemical and petroleum industries. Distillation will represent a large and continuing capital investment, and it will warrant continued research support.

## Acknowledgment

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