

DISTILLATION WITH VAPOUR COMPRESSION

An Undergraduate Experimental Facility

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FOR SOME PURPOSES it is useful to regard a distillation column as a heat engine, absorbing heat at a high temperature in the reboiler, performing separative work and discarding the heat at a lower temperature in the condenser.

The ideal separative work required to split a feed containing mole fraction x of component A and $(1-x)$ of B into pure components is then

$$W = -RT [x \ln x + (1-x) \ln (1-x)]$$

when the components obey Raoult's law. In practice for any real distillation, the efficiency

$$\eta = \frac{\text{ideal separative work}}{\text{heat into reboiler}}$$

is very small, commonly a few percent [1]. As a heat engine, a distillation column is very inefficient. And since a substantial amount of the chemical industry's energy use is in distillation, it is worth considering *why* the efficiency should be so low, and what can be done to improve it.

One reason for low thermal efficiencies is related

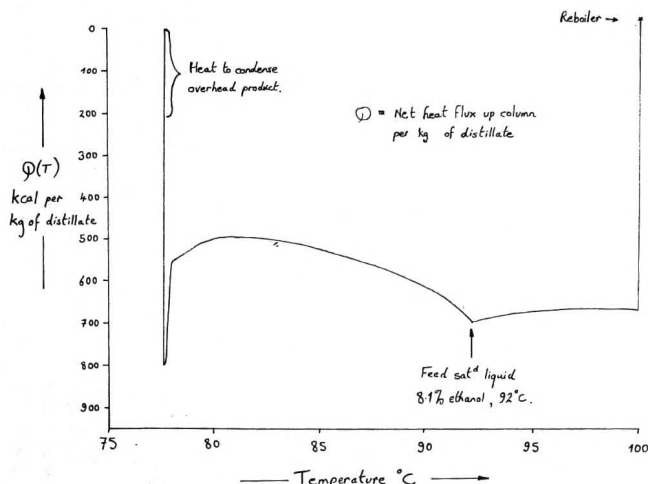
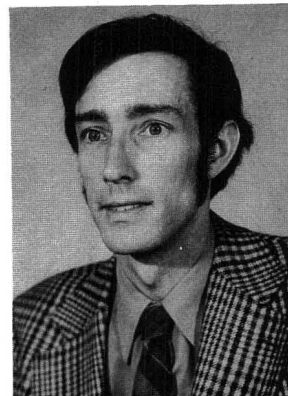


FIGURE 1. 'Q' Curve for ethanol-water at 1 bara



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to choice of reflux ratio: a reflux ratio is normally selected, or controlled, to obtain a desired purity in the column overheads. In many common separations (*e.g.* acetone-water, ethanol-water) the reflux ratio required in the last stages of rectification is far higher than that required just above the feed, or for the stripping section. Thus in order to perform a comparatively small amount of separative work for high-purity overheads, a great deal of heat has to be supplied at the reboiler temperature. If heat could be supplied at the (usually much lower) temperature at which it is required for separation, then higher thermodynamic efficiencies could be achieved.

'Q' curves such as Figure 1 are a graphic, though little-used, representation of heat flows in distillation. The construction is described in Flower and Jackson [2] and utilises the enthalpy-concentration relationships used in construction of the familiar Ponchon-Savarit diagram. Figure 1 reveals that separation of ethanol-water close to the azeotrope requires 800 kcal per kg of distillate, compared to only 215 kcal per kg required to condense the overhead product—equivalent to a minimum reflux ratio of $800/215 = 3.7$. But at a temperature only 1 degree C higher (75% w/w ethanol) the required net heat flow has fallen to 500

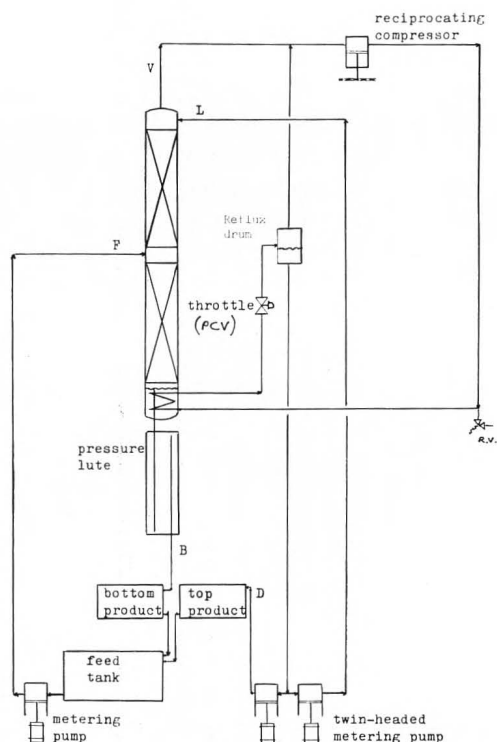


FIGURE 2. Schematic diagram of vapour compression still

kcal per kg distillate (minimum reflux ratio 2.3). Thus only a fraction of the heat needs to be transferred over the full temperature range between reboiler and distillate: much of the heat required could be transferred over a temperature difference of 1 degree C! This is a point often overlooked in considering energy savings in distillation, though it is of major significance in "difficult" separations such as ethanol rectification in which most of the separative work is achieved over a very small temperature range [3].

OVERHEAD VAPOUR COMPRESSION

It is in such systems that overhead vapour compression schemes can produce very large energy savings: because of the low temperature lift, the compressive work required is very small and this "heat pumping" system exhibits a high coefficient of performance. Overhead vapour recompression is the simplest means of transferring reject heat to a point where it can perform useful separative work. It is applied in a number of industrial separations—principally those involving close-boiling components such as propylene-propane [4], and is gaining importance as energy costs rise.

CHOICE OF EQUIPMENT

To demonstrate the principles of vapour compression distillation, two senior students were given a "de-

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sign and build" project. Since only six months were available for this, some key equipment had to be purchased in advance, and this effectively dictated the scale of the pilot plant which was constructed. At the heart of the system was the compressor, a Compton D 416-4 gas compressor with 316 stainless steel gas contacting parts, delivering 1.2 scfm (equivalent to 1.37 mol min⁻¹ at 30°C) at 30 psig. Stringent considerations of laboratory safety effectively restricted our choice of separation system to refrigerants—a fortuitous restriction since refrigerant mixtures are "well-behaved" on compression and many mixtures obey Raoult's law over a wide range of temperature, pressure and composition.

WORKING FLUIDS

In Edinburgh where laboratory temperatures in the six winter months average 60°F, the choice of refrigerants was unequivocal: Refrigerant 11 (CCl₃F) boiling at 77°F (25°C) and Refrigerant 113 (CCl₂F-CClF₂) at 119°F (48°C). In labs where the ambient temperature does not fall below 78°F, a mixture of R112 (freezing point 78°F, boiling point 198°F) with R113 could be used. However the low boiling mixture has the advantage that heat losses from the system are minimised: the rectification system in particular operates close to ambient temperature and does not suffer from the enormous internal reflux generated in most laboratory stills. (A disadvantage is that all storage vessels must be cooled by circulating water to avoid excessive losses; and all must be vented through a refrigerated trap.) The mixture may be analysed by densitometry or by gas chromatography, though special procedures had to be developed to ensure representative sampling. Figure 3 shows the thermodynamic cycle for pure refrigerant 11 as overhead, with adiabatic compression. The resulting coefficient of performance is derived from the enthalpies of vapour and liquid (Fig. 3)

$$\text{c.o.p.} = \frac{\Delta H_c}{W_T} = \frac{324 - 142}{324 - 302} = 8.3$$

One other critical choice concerns the method of pressure control. Computers are used throughout our laboratories for data acquisition and control, so this was the rational choice for the pressure control loop. Two pressures and twelve temperatures are read via

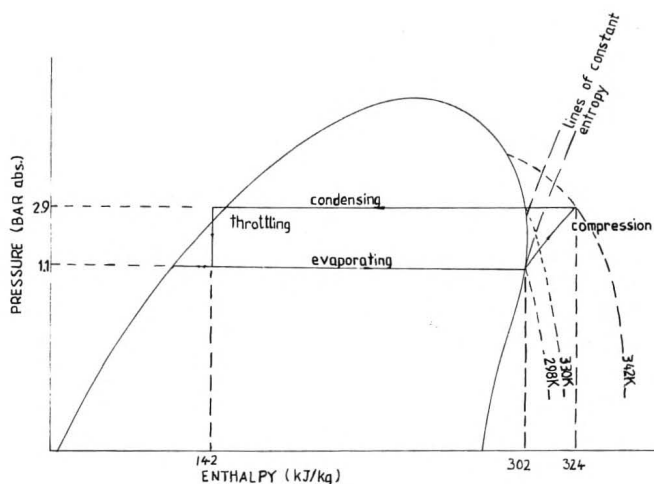


FIGURE 3. Thermodynamic cycle for heat pump working fluid

the IEEE port and a standard interface box, and the pressure control is effected through the user port, with a D/A converter providing 4-20 mA output.

FINAL DESIGN

The design finally evolved by the students is shown in Figure 2. The column is 3" diameter, 6' tall packed with 1/4" glass raschig rings with feed at the midpoint. The reboiler is a standard borosilicate glass unit of 5 ft² (0.5 m²) area with a nominal heat transfer coefficient of 50 BTU hr⁻¹ ft⁻² F⁻¹ (290 Wm⁻²K⁻¹). Auxiliary heating is supplied by electrical heating tape for starting only.

Design operating conditions are 1.1 bara in the still base and 2.9 bara for the compressed vapour, which thus condenses at around 57°C. This gives a 12 degree temperature difference across the condenser/reboiler, which is more than adequate for the heat duty of 730W (2500 BTU/hr).

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COMPRESSOR OUTLET: SETPOINT=2.9  BARA
VALVE OPENING= 68 % ACTUAL=3.0  BARA
CONDENSATION TEMP=53.0  DEGC
REBOILER TEMP=41.5  DEGC
ESTD HEAT TRANSFER RATE=1.67  KW
USEFUL COP OF SYSTEM=10.67

MF-R11  TEMP(C)  PRESS(BARA)
-----
REBOILER  .19  .35  41.5  0.996
BOTTOM PLATE .25  .43  39.8  0.996
FEED
OVERHEAD  .82  .91  27.9  0.996
FLASH VESSEL .86  .93  27.2  1.0

COMPRESSOR PERFORMANCE
-----
INLET      43 (DEGC) 0.996 (BARA)
OUTLET     58 (DEGC) 2.985 (BARA)
SUPERHEAT 15 (CDEG)
REFLUX RATIO SET TO TOTAL
CALCULATED HETP = 0.71 METRES
BANDWIDTH= 50 % RESET TIME= 20( SECS)
TO ALTER, HIT [A],[B],[C],[D], OR [E]

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FIGURE 4. Typical screen display

Flashing liquid from the pressure control valve is taken to a separator; the vapour is returned to the suction side of the compressor, while the liquid is proportioned to reflux and storage. The column mass balance is secured by an overflow lute system on the reboiler, which ensures constant reboiler level at pressures between 0.95 and 1.1 bara approx. The column pressure is controlled manually by cooling water flow to the reflux drum, which sets the vapour pressure.

Since the computer logs both temperatures and pressures it can compute composition at points in the plant where there is vapour-liquid equilibrium, using

$$x_A = \frac{P_T - p_B^*}{p_A^* - p_B^*}$$

and the Antoine equations for p_A^* and p_B^* .

These are displayed on the screen together with the pressure control variables, heat transfer rate, compressor performance and calculations of HETP (overall) and the C.O.P. of the system as a heat pump. Figure 4 is a typical screen display.

Obtaining representative samples of such volatile liquids for VPC is extremely difficult. Eventually on-line samples were drawn through capillary tubing into evacuated glass bulbs, from which samples of vapour admixed with air were withdrawn by gas syringe. This gave an independent check on the computed compositions, which were in reasonably close agreement.

USE IN TEACHING AND RESEARCH

The pilot plant was built in two months at modest cost, using standard components. The thermocouple interface was a later addition which, with the associated software, greatly enhances the teaching value of the rig. Freed from the necessity of carrying out tedious calculations, students can rapidly appreciate the effect of changing reflux ratio (for instance) on HETP and heat pump c.o.p. The compressor performance may also be evaluated over a range of compression ratios. The rig has proved its worth in both teaching and research and is currently being used in an investigation of difficult separations at low temperature lift.

A complete design report and specifications are available from the author.

REFERENCES

1. Freshwater, D. C., *Trans Inst Chem Engrs.*, 29, 149-160, 1951.
2. Flower, J. R. and M. A. Jackson, *Trans Inst Chem Engrs.*, 42 T 249-258, 1964.
3. Mostafa, H. A., *Can J Chem Engrs*, 59, 487-491, 1981.
4. Finelt, S., *Hydrocarbon Processing*, Feb. 1979, 95-98.

NOMENCLATURE

p_a^*, p_B^*	s.v.p. of components A, B
P_T	total pressure on system
R	gas constant $J \text{ mole}^{-1} \text{ K}^{-1}$
T	temperature
W	ideal work of separation, $J \text{ mole}^{-1}$
x_A, x_B	mole fraction of A, B in liquid
η	(1st law) efficiency of separation
ΔH	heat given up in cooling and condensing overhead vapour
W'	work done by compressor <input type="checkbox"/>

ChE book reviews

SCALEUP OF CHEMICAL PROCESSES

By Attilio Bisio and Robert L. Kabel
John Wiley and Sons, New York (1985) 18 Chapters,
699 Pages, \$69.95

Reviewed by
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Monsanto Company
Florissant, MO 63031

This is an impressive book and one that should be on the desk of everyone who teaches transport phenomena to chemical engineering students. The reasons are well stated in the first and last chapters of the book. From the first chapter, "Introduction to Scaleup," we have the quote defining scaleup as "The successful startup and operation of a commercial size unit whose design and operating procedures are *in part* based upon experimentation and demonstration at a smaller scale of operation." From the last chapter, "Scaleup: Overview, Closing Remarks, and Cautions," there is a classic line, "Indeed, to a very significant extent, scaleup *is* chemical engineering." The book does, in fact, describe what chemical engineers *do* and, for that reason, it will also be valuable to industrial people involved with developing and even troubleshooting processes and to those striving to teach potential chemical engineers how to design chemical processes and plants.

The subject matter is introduced by describing several processes and the pertinent experiences involved in developing a concept to commercial reality. This is followed by a general and well thought out treatise on the use and utility of mathematical models in the description of chemical processes. The core chapters cover the basic subjects of reactor design and development, flow and mixing, and mass transfer and separations. All of these chapters begin with a discussion of major issues pertinent to the topic. This

is followed by a discussion of fundamentals and the chapters conclude with practical issues covering commercial equipment, scaleup and uncertainties.

The last chapters cover practical matters, including a necessary chapter on environmental problems and the issues facing our industry today. People doing commercial process development acquire various "rules of thumb" from years of experience and Chapter 17, in this book, has several pages of these "rules of thumb." They make delightful reading.

The final chapter brings the book together with a discussion of the realities that are present in scaleup/chemical engineering.

The book had its genesis in a short course, taught by the authors over several years, and it is the only modern book on the subject. The 18 chapters were written by some 17 different practitioners of the art, but it reads as though it was written by an individual. The authors have done a remarkable job of organizing and integrating the chapters into a unified whole, while allowing each chapter to stand on its own.

The chapters and authors are:

1. Introduction to Scaleup, *A. Bisio*
2. Mathematical Modeling, *D. M. Himmelblau*
3. Reaction Kinetics, *R. L. Kabel*
4. Homogeneous Reaction Systems, *R. L. Kabel*
5. Reactors for Fluid-Phase Processes Catalyzed by Solids, *G. F. Froment*
6. Fluid-Fluid Reactors, *Y. T. Shah and W. D. Deckwer*
7. Selection of Reactor Types, *R. L. Kabel*
8. Flow Patterns and Residence Time Distributions, *E. B. Nauman*
9. Mixing Processes, *J. Y. Oldshue*
10. Fluidized Beds, *J. M. Matsen*
11. Laminar Flow Processes, *E. B. Nauman*
12. Stagewise Mass Transfer Processes, *J. R. Fair*
13. Continuous Mass Transfer Processes, *J. R. Fair*
14. Solid-Liquid Separation Processes, *L. Svarovsky*
15. The Environmental Challenges of Scaleup, *P. B. Lederman*
16. Evaluating Materials of Construction in Pilot Plant Corrosion Tests, *P. E. Krystow*
17. Gaining Experience Through Pilot Plants and Demonstration Units, *F. G. Aerstin, L. A. Robbins, A. J. Vogel*
18. Scaleup: Overview, Closing Remarks and Cautions, *G. Astarita*

This work should be a part of a chemical engineers training and while I would not suggest that it be a required text because of its cost, some way should be found to introduce young engineers to its contents.