ChE laboratory

BASIC CHEMICAL ENGINEERING EXPERIMENTS

W. E. JONES University of Nottingham Nottingham, United Kingdom NG7 2RD

This article is concerned with laboratory work undertaken in the early years of an undergraduate chemical engineering course and is based on the author's experiences at the University of Nottingham. Our department teaches two undergraduate courses in parallel: a six-semester BEng and an eight-semester MEng. Each semester is of fifteen weeks duration, and there are two semesters per academic year. The first four semesters are common, and it is to this portion of the course that this article is devoted.

The objectives of any student's experimental work are:

- to illustrate and reinforce chemical engineering theory and principles
- to provide hands-on experience with some commonly used process equipment
- to demonstrate experimental methodology and techniques

Further time available for laboratory work is limited.

To meet the above objectives, a wide range of experiments is required, and those used at Nottingham in the first four semesters are summarized in Table 1. The experiments must be tightly defined and carefully arranged so that a two-student team can perform the work in a three-hour session. Groups of three to six experiments are associated with appropriate modules, and in most cases students will undertake one or two experiments from this group as an integral part of the module.



Warren Jones holds BSc and PhD degrees in Chemical Engineering from the University of Nottingham and is a registered Chartered Engineer. He has a wide-ranging interest in both front-end process and detailed plant design developed initially through nine years of experience with a major engineering and construction company. Teaching responsibilities include several design courses, process economics, and engineering thermodynamics. Some of the experiments listed in Table 1 are standard and are found in many undergraduate laboratories. Others are "classic" chemical engineering experiments, and yet others introduce unusual aspects to the laboratory. It is not the author's intention to describe every experiment in the table, but rather to take selected experiments and discuss

- how some of the standard experiments can be made more interesting
- how some of the "classic" experiments can be performed more effectively
- how unusual aspects can be exploited in the laboratory

LABORATORY PHILOSOPHY

The experiments are all manually controlled. This forces the students to "work the rig" and develop a feel for the way a process plant behaves. They learn that flow and pressure systems respond very quickly and hence large quantities of data can be collected very quickly, but that heat and mass transfer systems are much slower and may require considerable time to reach steady state. Some process interactions are found, *e.g.*, many students are initially surprised when steam pressure drops on increasing cold-side flow through a heat exchanger.

Nottingham's laboratory has its own boiler, and steam is distributed to most of the rigs that require heat input. This is in line with our policy of trying to emulate industrial practice and is an important element in mimicking the behavior of a "real" process plant.

Many of the experiments are designed and built with a significant glass and/or acrylic resin (perspex) content so that students can see the process operating. This is particularly important for situations such as vapor-liquid contacting; boiling, and two-phase flow. However, care is needed because poor thermal conductivity (as compared to copper or steel) can distort the heat transfer phenomena being investigated.

© Copyright ChE Division of ASEE 1993

FLUID MECHANICS

Centrifugal Pump Operation • Since centrifugal pumps are widely used in process plants, it is important for students to become familiar with their performance characteristics. The simplest experiment, commonly performed, is measurement of head-capacity characteristics. (As an aside, it is worth considering how few student textbooks mention the headcapacity curve, let along explain how it is used.) However, several interesting variations on the basic experiment are possible.

The first version measures head and shaft power as a function of capacity at three speeds of rotation. The speed variation is obtained by using a belt and pulley drive from a fixed-speed electric motor. Belt and pulley drives have significant friction effects, and it is important to "back out" these losses when determining the pump shaft power. To do this, the pump must be capable of being disconnected and the remainder of the system run with no load to determine a base power consumption. The shaft power is then obtained as the difference between measured power consumption of the motor in normal operation and the base power consumption.

After measuring the head and shaft power, the pump efficiency is easily calculated, and the students are able to plot the three characteristic curves of the pump: head, shaft power, and efficiency versus capacity, all with lines of constant speed. A further

FLUID MECHANICS Venturi tube* Pipe flow friction* Orifice discharge* Velocity profiles in a pipe Pressure drop in pipe fittings Liquid distribution Pump operation at varying speed Pumps operated in parallel and series Pump operated on various fluids Air lift pump

MASS TRANSFER Measurement of gas diffusivity (stagnant film) Measurement of gas diffusivity (counter-diffusion) Wetted wall column Wetted disc column

ENGINEERING THERMODYNAMICS

Turbine isentropic efficiency Fan polytropic efficiency Refrigeration COP

PARTICLE MECHANICS Packed and fluidized beds (air) Packed and fluidized beds (water) Particle elutriation

TABLE 1

HEAT TRANSFER

Heat transfer coefficient inside tubes Heat transfer coefficient outside tubes Double pipe heat exchanger Plate heat exchanger Unsteady-state tank heating Boiling heat transfer Thermosyphon reboiler Heat transfer in fluidized bed Unsteady-state heating of solids Temperature profiles in solids

UNIT OPERATIONS**

Packed column distillation Oldershaw column distillation Adiabatic humidification Wet solids drying Liquid-liquid extraction (mixer-settler)

CHEMICAL PROCESS PRINCIPLES Boiler efficiency Mass and heat balances on internal combustion engine Heat balance on batch process

Heat balance on recycle process *Compulsory experiment

**Experiments take two three-hour sessions

feature of this experiment is that it may be used as another illustration of dimensionless correlations.^[1] In particular, the collapse of the various head-versuscapacity lines due to speed variation into one line on dimensionless coordinates is very effective.

In some large pumping operations it is necessary to run two or more pumps in parallel (*e.g.*, cooling water circulation), and this provides an idea for another useful centrifugal pump experiment. Two pumps are run individually, in series and in parallel. Students measure the head-capacity characteristics for each configuration and learn how individual pump characteristics can be combined to predict the headcapacity characteristics for series and parallel operation.^[2] If the two pumps are dissimilar, then the limitation imposed by the lowest shut-off head of the pair again surprises students.

The third centrifugal pump experiment is simpler and demonstrates the important characteristic that a given pump will always throw up the same head at a given capacity, even though it is pumping different liquids, provided liquid viscosity is low. (This explains why water testing by a pump manufacturer is often sufficient.) Hence, as the liquids pumped have different densities, the pressure increase measured across the pump changes. Measuring the head-capacity characteristics of a pump on two different fluids (*i.e.*, water and kerosene) is a good way to demonstrate this point.

> Fluid Distribution • One of the most neglected areas of chemical engineering is fluid distribution. The operation of many real plant pieces can be impaired by mal-distribution, and the requirements to obtain approximately uniform distribution are quite restrictive.^[3,4] An experiment to illustrate the difficulties studies one branch of a pipe distributor. A variety of branches fabricated from 22mm ID pipe, each 1.0-m long, are available. Each branch has six uniformly spaced discharge holes on the bottom, and four (also uniformly spaced) pressure tappings on the top. The branches differ in discharge-hole sizes, which range from 5.6 mm to 10.3 mm. Each branch may be connected in turn to a constant head supply of water with the throughput controlled by a throttling valve in the supply line.

When they first run the rig, most students are caught unaware by the fact that the pressure *increases* along the branch and the increase is relatively more marked for the larger-size discharge holes. This phenomena is easily explained by application of Bernoulli's equation and is the cause of the mal-distribution problem. The higher pressure at the closed end of the branch causes a greater discharge rate there as compared to the inlet.

Students determine the water discharge rate from each hole and record the pressure profile along the branch for a few throttle valve settings. The procedure is then repeated for other branches, and this collected data allows the students to infer the requirements for good distribution in terms of water inlet velocity to the branch and pressure drop across the discharge holes. Friction loss along the branch is not significant in this rig. The criterion used for good distribution is that the maximum variation between highest and lowest hole discharge rates for a given throttle valve setting should be less than 10%.

Two-Phase Flow • Two-phase flow occurs frequently in process plants, and incorrect analysis or neglect may result in poor plant performance or hazardous situations.^[5] It is often an area not covered in undergraduate courses, and suitable student experiments that can be performed in three hours are rare. The easiest way to introduce a two-phase flow element into the laboratory is with an air-lift pump.^[6] The rig is based on a nominal 25-mm ID glass riser, and liquid lifts of up to 3.33 m are used. Motive air may be injected at submergences up to 5.5 m, and students are asked to investigate the variation in water throughput as the air flowrate is changed. The water throughput passes through a sharp maximum illustrating the gravity- and friction-dominated regimes at low and high air flowrates, respectively.

HEAT TRANSFER

Effect of Velocity • The fundamental experiments in this group involve measurement of inside and outside tube heat transfer coefficients. The simplest and most effective way to perform both of these measurements is to ensure that the resistance of the coefficient to be measured is the dominant effect. Hence, the inside-tube heat transfer coefficient is measured for low-pressure air flowing through a copper tube and heated by condensing steam in a surrounding jacket. Likewise, the outside-tube heat transfer coefficient is obtained by blowing low-pressure air over the "shell side" of bundles comprising copper tubes arranged on square and triangular pitches; heating is again by condensing steam, but this time it is contained in the tubes. The heat transfer resistance provided by condensing steam and copper tubing is negligible compared to the air-film 54

resistance, and the measured overall heat transfer coefficient is a very good approximation to the airfilm coefficient. Both experiments will produce straight line plots of Nusselt number versus Reynolds number on log-log axes.

Our experience indicates that the best technique for demonstrating the effect of velocity on a liquid film heat transfer coefficient is construction of a Wilson plot.^[7] This approach is used because assuming the measured overall heat transfer coefficient approximates the individual film coefficient is not sufficiently accurate when heating a liquid. The Wilson plot takes the condensing steam, copper wall, and dirt resistances as constant, and hence the reciprocal of the measured overall coefficient can be plotted as a straight line against the reciprocal of (process water flowrate)^{0.8} if we assume that the Dittus-Boelter equation models the water film coefficient inside a tube. The experiment is performed in a steam-heated double-pipe exchanger with condensing steam in the annular space. Extrapolation of the straight line to its intercept with reciprocal of the measured overall coefficient axis gives an estimate of the sum of all the other resistances to heat transfer. Using a typical condensing steam film resistance (not very significant) and calculating the resistance due to the copper wall means the only unknown resistance, sum of the dirt films, can be determined.

The advantage of performing the heat-transfer experiments as described above is that it avoids measuring the metal-wall temperature which can be unreliable and consume considerable technician time in dismantling the equipment to investigate suspect readings.

Condensate Blanketing and Sub-Cooling • The tank-heating experiment compares warming a batch of liquid using a submerged coil in the tank with a pumparound through an external heat exchanger. The heat transfer area of the coil and exchanger are equal so for the same steam operating pressure it is interesting to observe that the tank may be heated faster or slower than by the coil, depending on the pumparound through the exchanger. Applying the equations in Perry and Green^[8] it is possible to determine the overall heat transfer coefficients for the unsteady-state heating process in each case. However, the overall heat transfer coefficient determined for the submerged coil tended to be unreproducible and lower than expected.

As originally built, the submerged coil used 6-mm diameter tubing with a large number of turns. Excessive condensate "hang up" in the coil, obscuring the heat transfer area, was suspected, and therefore

Chemical Engineering Education

the coil was replaced by a harp using vertical tubes of 13-mm diameter tubing. The condensate now drains freely and the measured overall heat coefficient is larger, in line with the anticipated value, and reproducible.

An extreme example of obscuring the heat transfer surface by condensate was encountered in the plate heat exchanger unit. The very good heat transfer performance of the plate heat exchanger means that the steam is not only condensed but highly sub-cooled



 T_s = steam saturation temperature at operating pressure T_c = steam condensate exit temperature

 $T_1, T_2 =$ process water inlet/outlet temperature

m = process water flowrate

Estimate of cross-over temperature

$$\mathbf{T} \approx \left(\frac{\mathbf{h}_{water}(\mathbf{T}_{s}) - \mathbf{h}_{water}(\mathbf{T}_{c})}{\mathbf{h}_{steam}(\mathbf{T}_{s}) - \mathbf{h}_{water}(\mathbf{T}_{c})}\right) \left(\mathbf{T}_{2} - \mathbf{T}_{1}\right) + \mathbf{T}_{1}$$

Log-mean temperatures are given for the condensing and sub-cooling regions by

$$\Delta T_{c} = \frac{T_{2} - T'}{\ell n \left(\frac{T_{s} - T'}{T_{s} - T_{2}}\right)} \quad \text{and} \quad \Delta T_{sc} = \frac{\left(T_{s} - T'\right) - \left(T_{c} - T_{1}\right)}{\ell n \left(\frac{T_{s} - T'}{T_{c} - T_{1}}\right)}$$

Define

$$(UA)_{c} = \frac{Q_{c}}{\Delta T_{c}}$$
 and $(UA)_{sc} = \frac{Q_{sc}}{\Delta T_{sc}}$

Weighted temperature difference

$$\begin{split} \Delta T_{w} &= \frac{Q_{c} + Q_{sc}}{\left(UA\right)_{c} + \left(UA\right)_{sc}} \end{split}$$

Weighted overall heat transfer coefficient
$$U_{w} &= \frac{Q_{c} + Q_{sc}}{\Delta T_{w}A} = \frac{\left(UA\right)_{c} + \left(UA\right)_{sc}}{A} \end{split}$$

Figure 1.

Winter 1993

to a temperature near that of the cold inlet process water to be heated. The plate heat exchanger is arranged for parallel flow, i.e., strictly counter-current operation, but the heat transfer process deviates substantially from the commonly analyzed example in undergraduate courses. Figure 1 clearly illustrates the discontinuity in the enthalpy-versustemperature plot which is the source of the difficulty. Analysis of the experimental results is difficult because the heat transfer areas for condensing and sub-cooling cannot be identified. The best approach is to determine (UA)_c and (UA)_{sc} based on an estimate of the process cross-over temperature, T', as indicated in the figure. The performance of the unit can then be expressed in terms of the weighted temperature difference, ΔT_w , and weighted heat transfer coefficient, U_w .^[9] This experiment is important in illustrating to students that you simply do not take the four terminal temperatures of a heat exchanger to calculate a log mean temperature difference without considering the process actually occurring in the unit.

Boiling Heat Transfer • One area of heat transfer where visual inspection is important is boiling. We use a proprietary rig for demonstrating nucleate and film boiling and for measuring heat flux variation with temperature difference. This works well, but industrial reboilers exhibit much more complex phenomena. The choice generally lies between vertical and horizontal thermosyphons. If a vertical thermosyphon (where boiling occurs in the tubes) is selected for laboratory work, then a heavy duty glass tube is required in order to see the boiling and the thermal resistance of the wall dominates the heat transfer. Hence, for the laboratory, boiling on the outside of copper tubes in a horizontal thermosyphon is a better choice. This construction means that condensing steam is contained in the Utube bundle and flow boiling phenomena can be inspected through a glass shell.

The experiment most suitable for second-year students is the measurement of overall heat transfer coefficient variation with shell-side flow through the unit. But a word of caution is required this type of unit (both laboratory- and commercialscale) commonly exhibits an instability at low loads.^[10] In fact, an excellent third-year laboratory project is to determine the stability envelope for the unit in terms of the two parameters, steam pressure and shell-side flow.

Fluidized Bed Heat Transfer • Heat transfer from a hot surface to a fluidized bed is worth including because the heat transfer coefficient passes

through a maximum as fluidizing air flow is varied. The rig comprises a vertical copper tube (heated by condensing steam) located at the center of a small fluidized sand bed. Again, the measured overall heat transfer coefficient will be a good approximation to the film coefficient of interest. The heat transferred is based on the gain in air temperature between the bed inlet and outlet. In addition, a few thermocouples are located in the bed to demonstrate the very effective mixing which occurs and the resulting uniformity through the bed. But do not place the lowest of the bed thermocouples closer than about 25 mm to the bed support because there is a very rapid temperature transition in this region.

Ideally, students should investigate the heat transfer coefficient versus air-flow curve for various fluidized solids. Unfortunately, the rig is slow to stabilize at new conditions, and readings on only one fluidized solid are possible in the period of three hours. Correlations for heat transfer between hot surface and fluidized bed tend to be problem specific and not very accurate, so the experiment simply asks students for a comparison of the measured maximum heat transfer coefficient with that predicted by the equation of Zabrodsky, Antonishin, and Parnas.^[11]

Heat Losses • Finally, the quality of experimental results can be significantly affected by the way measured data is processed. This is particularly true of heat transfer experiments where the small scale means the rigs have a relatively high surface-tovolume ratio leading to high heat losses. This problem is compounded in some cases by the fact that rigs are left at least partially unlagged for visual inspection of the process. Good engineering practice means that students are encouraged to check the consistency of their data by comparing heat gain by the process stream with heat released from the steam condensate collected. But students need to consider which heat load to use in the calculation of heat transfer coefficients. As a rule of thumb, if a condensing steam is involved the heat losses will be large and it is best to base the calculations solely on the process heat gain. If two streams, both at modest temperature, are exchanging heat, then the average of heat gain and heat lost is likely to be the best assumption.

MASS TRANSFER

The major problem with mass transfer (and for that matter, unit operations) is that it involves composition measurement. Compared to temperature, pressure, and flow measurements, composition measurement is much more time consuming and offers a greater potential for error. Thus it is worth expending some effort to devise experiments which either automate the composition measurement or use materials for which a simple physical measurement can be used to infer the composition.

Gas Diffusivities • The measurement of gas diffusivity in the context of a stagnant film by Stefan's method is an excellent experiment as it avoids direct composition measurement. In the case of counter-diffusion, we use Gover's method^[12] modified by intro-duction of a tracer gas to simplify the GLC analysis.^[13] The use of a tracer gas means that all concentrations of diffusing component can be measured relative to the tracer and thus avoids absolute concentration measurement, which is helpful.

Film Mass Transfer Coefficients • The wettedwall column^[14] for investigating gas-film mass transfer coefficients has a reputation as a difficult experiment to make work with respect to establishing and maintaining a wetted wall. Most of the difficulty is due to the initial liquid distribution. The technical staff at Nottingham has devised a distributor which allows a stable film to be easily established at the start of a three-hour session and just as easily maintained throughout the period (see Figure 2). The only maintenance needed is cleaning of the small-diameter holes which can foul. The film produced normally has a slight ripple (as one might expect at slightly higher liquid loadings), but the data collected by students is sufficiently good to demonstrate that the Sherwood number is proportional to the (Reynolds number)^{0.8}. The rig is run on water and air, and the inlet and outlet gas compositions are easily measured by a humidity probe utilizing electrical capacitance.

To investigate liquid-film mass transfer coefficients, we use a wetted disc column^[15] and strip dissolved oxygen out of water using nitrogen.^[16] The advantage of this system is that oxygen compositions can be easily measured by using a dissolved oxygen meter and the quantity stripped is so small that the gas phase may be treated as pure nitrogen.



Figure 2. Distributor for wetted-wall column Chemical Engineering Education

UNIT OPERATIONS

Distillation • The small scale distillation columns are run on a methyl ethyl ketone-toluene mixture which has the advantage that compositions are easily inferred by refractive index measurement.^[17] The columns are run at total reflux, and the liquid from the condenser and bottom of contacting section are sampled to determine separation performance.

Students investigate the effect of reboiler heat input, *i.e.*, effectively changes vapor-liquid traffic in the column, on the separation achieved. To obtain measurable changes in distillate composition it is necessary to carefully select the composition of the mixture initially charged to the reboiler. A mixture comprising 90% toluene/10% methyl ethyl ketone is about right and will give distillate compositions in the range of 80-95% methyl ethyl ketone provided excessive separation capability is not installed (see below). Distillate compositions approaching 98%-plus of methyl ethyl ketone should be avoided.

The main problem areas of distillation experiments are heat losses and column end effects. Columns of 50-mm diameter are the very minimum to use. Despite being well insulated or vacuum jacketed, heat losses are significant, and the vapor-liquid traffic changes through the column. Thus the separation performance variation with heat input measured is at best indicative. In the case of packed columns, heat losses help accentuate the tendency for liquid channeling along the wall. Using a column



Figure 3. Packing support—simplification of a commercial injection plate.

diameter of 50 mm means that a column diameterpacking size ratio of 8 can be achieved, and this helps counter channeling.

End effects are mass transfer occurring outside the contacting section of the apparatus, *e.g.*, mass transfer between liquid leaving the bottom of the contacting section and vapor rising from the reboiler. These effects can be minimized by placing the liquid sample points close to the top and bottom of the contacting section.

There is an additional end effect which is easily introduced into packed towers by using inappropriate packing support. If the vapor and liquid are not routed separately through the packing support, a liquid pool forms above the support—and the vapor must bubble through this pool. This arrangement effectively adds the equivalent of one theoretical plate to the height of packing, it distorts mass-transfer performance, and it is potentially dangerous because it induces premature flooding.

Figure 3 shows the preferred arrangement. It allows the ascending vapor to enter the packed bed through chimneys while the descending liquid drains through holes in the support plate deck. The chimneys should occupy 15-20% of the cross-sectional area, and the hats prevent liquid from draining down the inside.

The unit operations experiments require two afternoons (see Table 1). In the case of packed column distillation, two separate packed heights are investigated (one on each afternoon): 23 cm and 46 cm of 6mm ceramic Berl saddles. If excessive end effects are avoided the students should be able to confirm that roughly twice the number of transfer units are available in the longer bed compared to the short one. The Oldershaw column distillation also takes two afternoons and investigates columns containing 3 and 5 actual plates, but the experiment provides an interesting detective challenge for the students.

The Oldershaw plates in the two columns are identical: same diameter, same number of holes, and same hole size. The three-plate column is correctly assembled and exhibits a plate efficiency of approximately 70%. But the measured separation of the fiveplate column is not proportionately better, and students are asked if they can see any factors leading to the deterioration in performance.

The five-plate column is incorrectly assembled. The plates are not level, so only part of the plate bubbles and the downcomer is badly located—with the result that much of the plate is short-circuited. The experiment very effectively points out the need for proper vapor and liquid contacting.

Mass and Heat Transfer in Packed Towers • A 57 simple experiment illustrating the influence of gas and liquid rates on gas phase transfer coefficients in packed towers is based on adiabatic humidification^[18] using the air-water system.

Water is circulated over the packed bed in a pumparound system and it stabilizes at the adiabatic saturation temperature. Obviously, there is no liquid-phase mass or heat transfer resistance, so all the resistance lies in the gas film and the problem of interface condition has been eliminated. Compressed air (hence, of very low humidity) is pressure-reduced to just above atmospheric, and then heated to near 100°C prior to counter-current contact with the circulating water. Contacting is over a short depth of relatively open packing: 125 mm of 30-mm metal Intalox Saddles. As before, use of the air-water system means composition measurements can easily be made by using a humidity probe.

The driving force for heat transfer is the temperature difference between the air and circulating water at any elevation. Likewise, the mass-transfer driving force is the difference between the saturation humidity at the water temperature and the actual humidity of the air. If the packing depth is too great or the air flow is too low, these driving forces will go to zero at the top of the packing. It is therefore important to limit the packing depth and use high air flows in order to obtain measurable driving forces. Obtaining suitable air flows without flooding requires use of a relatively open packing, but packing wetting is then never completely satisfactory. The experimental results are particularly sensitive, as one might expect, to the condition of the humid outlet air.

The mass and heat transfer coefficients may be correlated by equations of the form^[18]

 $h_Da = C_DG^mL^n$ and $h_Ga = C_GC^pL^q$

where (h_{Da}) and (h_{Ga}) are the volume-based mass and heat transfer coefficients, G and L are the gas and liquid loadings on the packing, and C_D and G_G are constants. Hence, by collecting a grid of measurements comprising four gas loadings by four liquid loadings it is possible to plot on log-log axis (h_{Ga}) and (h_{Da}) versus G at constant L, or L at constant G. In the time available, the values obtained for m, n, p, and q are not particularly accurate (*e.g.*, m and p range 0.5 to 0.9, and n and q range -0.1 to 0.4), but the experiment clearly illustrates the strong dependence on G and weak dependence on L of gas-phase transfer processes in packed towers. It is also useful to illustrate adiabatic saturation temperature and as practice in heat balancing over the tower.

Liquid-Liquid Extraction • Work is currently underway on a small mixer-settler experiment for 58 liquid-liquid extraction. Mass transfer is from kerosene to water, with kerosene drops dispersed in water as the continuous phase. This makes it easy to recycle kerosene. (The water contains a secondary haze and should not be disposed of directly to a sewer.)

The equipment is largely fabricated from glass because wall coalescing effects are minimized when using water as the continuous phase. Further advantages are visual inspection and easy maintenance.

The experiment has a number of unusual features. One is that the component transferred is methyl red indicator which changes color from yellow to red upon transfer from the kerosene phase to water (actually 0.01-M sulphuric acid). The color intensity in either phase can be related to concentration, and hence the phases can be sampled and subjected to colorimetry to determine the concentration of methyl red. Mixer samples need to be taken through hydrophobic and hydrophilic probes. Use of kerosene-water-methyl red is based on previous experience with this system in a pilot-plant scale liquid-liquid extraction unit.^[19]

The second unusual feature is that the apparatus allows the choice of two types of settler. A vertical settler^[20,21] is used on one afternoon, and a horizontal unit^[22] is used on the second. So in addition to having the potential to investigate the effects of such variables as throughput and mixer speed on stage efficiency, students can also study the effect of throughput on coalescing band depth in the vertical settler and coalescing wedge length in the horizontal unit.

A few words of warning on the use of methyl red are necessary. The properties of kerosene can vary slightly, and so the equilibrium line for distribution of methyl red between the kerosene and water phases has to be checked occasionally. Also, commercial methyl red should be recrystallized to improve purity before dissolving it in kerosene.

SUMMARY

This article has presented an overview of Nottingham's approach to basic experimental work in the laboratory. In addition to commonly performed experimental work, a number of novel experiments and unusual approaches have been adopted that both emphasize the practices and highlight the problems of industry. The author realizes that some of the descriptions are brief and would be pleased to answer any follow-up questions or hear of other academics' experiences with similar experiments.

REFERENCES

- 1. Kay, J.M., and R.M. Nedderman, *Fluid Mechanics and Trans*fer Processes, 1st Ed., Cambridge, 273 (1985)
- Karassik, I.J., W.C. Krutzsch, W.H. Fraser, and J.P. Messina (Eds.), *Pump Handbook*, 1st Ed., McGraw-Hill, New York, 2-170 (1976)

Chemical Engineering Education

- 3. Senecal, V.E., Ind. Eng. Chem., 49, 993 (1957)
- Perry, R.H., and D. Green (Eds.), Perry's Chemical Engineers' Handbook, 6th Ed., McGraw-Hill, New York, 5-48 (1984)
- Tilton, J.N., and A.W. Etchells, *The Chemical Engineer*, No. 465, 3 (1989)
- Perry, R.H., and C.H. Chilton, *Chemical Engineers' Handbook*, 5th Ed., McGraw-Hill, New York, 6-14 (1973)
- Coulson, J.M., and J.F. Richardson, *Chemical Engineering*, Vol. 1, 4th Ed., Pergamon, 410 (1990)
- Perry, R.H., and D. Green, (Eds.), *Perry's Chemical Engineers' Handbook*, 6th Ed. McGraw-Hill, New York, 10-38 (1984)
- Ludwig, E.E., Applied Process Design for Chemical and Petrochemical Plants, Vol. 3, 2nd Ed., Gulf, 50 (1983)
- Kister, H.Z., Distillation Operation, 1st edn., McGraw-Hill, New York, 451 (1990)
- Zabrodsky, S.S., N.V. Antonishin, and A.L. Parnas, Can. J. Chem. Eng., 54, 52 (1976)
- 12. Gover, T.A., J. Chem Educ., 44, 409 (1967)
- 13. Paterson, W.R., Chem. Eng. Ed., 19, 124 (1985)
- Gilliland, E.R., and T.K. Sherwood, Ind. Eng. Chem., 26, 516 (1934)
- Stephens, E.J., and G.A. Morris, *Chem. Eng. Prog.*, 47, 5, 232 (1951)
- Sherwood, T.K., and F.A.L. Holloway, *Trans. Am. Inst. Chem.* Eng., **36**, 39 (1940)
- 17. Steinhauser, H.H., and R.R. White, *Ind. Eng. Chem.*, **41**, 2912 (1949)
- Hensel, S.L., and R.E. Treybal, Chem. Eng. Prog., 48, No. 7, 362 (1952)
- 19. Hills, J.H., private communication
- Ryon, A.D., F.L. Daley, and R.S. Lowrie, *Chem. Eng. Prog.*, 55, No. 10, 71 (1959)
- Gondo, S., and K. Kusunoki, *Hydrocarbon Processing*, 48, No. 9, 209 (1969)
- 22. Hanson, C., (Ed.) Recent Advances in Liquid-Liquid Extraction, Pergamon, 544 (1971)

REVIEW: Staged Operations

Continued from page 27.

three chapters. Chapter 7 details the tedious hand calculations performed for the design of multicomponent systems. This provides motivation for the development of a computer code to implement the methods of Lewis-Matheson and Thiele-Geddes in Chapter 8 and the shortcut methods derived by Fenske, Underwood, and Gilliland in Chapter 9.

Complex distillation methods which handle azeotropic, extractive, and two-pressure distillation are covered in Chapter 10. Chapter 11 presents a switch from continuous operation to methods associated with batch distillation using the Rayleigh equation. Chapter 12 contains information regarding staged column design including column diameter, efficiency, and tray size, while packed column design is discussed in Chapter 13. Consideration of economic trade-offs between various design options leads to a more detailed discussion of economics in Chapter 14, which addresses such topics as capital and operating costs, energy conservation, and column sequencing.

The processes of absorption and stripping are ana-Winter 1993 lyzed in Chapter 15, using the Kresmer equations as well as the McCabe-Thiele method. Immiscible extraction in Chapter 16 is also discussed in the context of the McCabe-Thiele analysis. For extraction involving dilute mixtures, the Kresmer equations are shown to be appropriate. Example calculations for crossflow operations are also included.

In Chapter 17, generalizations of the McCabe-Thiele analysis are applied to washing, leaching, supercritical fluid extraction, and three-phase systems. Table 17.1 effectively summarizes the relationships between the various separation processes in terms of McCabe-Thiele analysis.

Extraction for partially miscible systems with triangular diagrams and the lever rule is analyzed in Chapter 18, and Chapter 19 introduces mass transfer concepts for design of packed beds, mass transfer coefficients, sum of resistances, and tray efficiencies leading to HTU-NTU analysis for absorbers and strippers. Note that rate-limited processes, including adsorption, chromatography, electrophoresis, and membrane separations, are included in a sequel to this text entitled *Mass Transfer Limited Separations*.

The predominant concept in this text is the McCabe-Thiele analysis. Distillation, absorption, and extraction processes are treated in terms of McCabe-Thiele diagrams. This approach emphasizes the analogies between these separation processes, which is very instructive from a pedagogical point of view. But it consequently results in a very narrowly focused book. The narrow focus is my major criticism of this text. Our current curriculum combines a study of equilibrium-staged separation processes with fundamentals of mass transfer and rate-controlled separation processes within a single course during the second semester of the third year. This text would only cover about one-third of the material for the course and therefore would require either additional texts or other supplementary material for the remaining topics. Since students are generally opposed to purchasing more than one text for any given course, I see this book filling the role of a supplementary text to complement a primary text that covers a broader range of material. The other alternative would be to devote an entire course to equilibrium-staged operations, for which this book would be an excellent text. However, with the chemical engineering curriculum already overloaded, it is difficult to justify creation of a new course for this single topic.

One additional minor criticism regards the appearance of the print. The type is not particularly easy on the eye since the book has evidently been produced in camera-ready form on a low-resolution (by printing standards) printer. \Box