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Innovative Chemical Engineering Teaching Experiments

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Engineering Experiments designs, manufactures and sells high quality teaching experiments for chemical engineering and related disciplines. Supporting materials include student handouts. Nine experiments cover separations, process dynamics and control, transport and flow, and reaction kinetics. Several experiments involve modeling and parameter estimation. Customized versions are available.

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Please contact us at spencer@columbia.edu for more details on the experiments and for price and delivery, or call 201-264-3511.
Packed-Bed CO₂ (or NH₃) Absorber

Model xxpak01

Packed bed absorber, showing air, CO₂, and water/NaOH feed lines, MSA CO₂ analyzer, terminal board for A/D board, Dwyer differential pressure cell, water manometer, CO₂, liquid and air rotameters, stepper-motor driven liquid flow control valve, Arrick stepper control box, 4 inch ID borosilicate column with Raschig ring packing, movable liquid outlet line with conductivity cell, three thermocouples in packing, conductivity meter, tracer injection points. NaOH feed tanks and pump not shown. An NH₃ absorber is also available.

**Packed-Bed CO₂ (or NH₃) Absorber:** The apparatus consists of a 4” ID borosilicate column containing about 30 inches of ceramic Raschig ring packing. A 0.5 N NaOH solution (or water) is fed to a distributor at the top of the column. Rotameters are used to measure the air, CO₂, and liquid feed rates. Two 20 L polyethylene tanks and a centrifugal pump supply the NaOH feed. The liquid flow to the top of the column is controlled by a needle valve, this valve driven by an Arrick stepper motor controlled by a computer program. The same experiment, but using ammonia and no NaOH, is also offered.
Instrumentation includes a Dwyer stainless differential pressure transducer used, in addition to a water manometer, to measure the pressure drop across the packed column. A conductivity cell mounted in the liquid effluent line is connected to a Cole-Parmer conductivity meter and measures the transient tracer level following tracer injection above or below the packing. An MSA infrared analyzer measures the CO₂ concentration in the effluent air. Three thermocouples (optional) may be mounted in the packing, but show very little temperature effect due to CO₂ absorption. A NI A/D board digitizes all signals, under control of LabVIEW programs. A computer (not included) is used for data acquisition and control.

The experiment operates in four modes, namely:

- The pressure drop across the column is measured by the differential pressure cell and the manometer as a function of the air and water flow rates. The results can be compared to theory, and flooding behavior can be demonstrated. Each run takes about ten seconds.

- The computer acquires transient effluent liquid tracer levels following conductive tracer injection above and then below the packing. A LabVIEW program calculates mean residence times and then the volume of water held up on the packing. The effects of air and water flow rates on hold up can be determined, and the onset of flooding demonstrated. Each run takes a few minutes.

- The steady state effluent CO₂ level is measured at selected CO₂, air and NaOH feed rates. The data can be processed to yield measures of the CO₂ absorption rate, for example the height of a transfer unit.

- A PID control algorithm is used to control the effluent CO₂ level by varying the NaOH feed rate via the stepper motor driven valve. Students can determine the effect of the controller gains on the controller performance. Stable and oscillatory behavior can be demonstrated.

The four modes of this comprehensive experiment can easily occupy two lab periods. Please contact us at spencer@columbia.edu for more details on the experiment, and for price and delivery.
**Catalytic Hydrolysis of Ethyl Acetate**

*Model xxact01*

Catalytic hydrolysis of ethyl acetate experiment, showing acetate feed tank, magnetic drive feed pump, acetate rotameter, stainless heat exchanger, jacketed glass reactor with ion exchange resin, flow control valve, flask for effluent sample collection, buret for sample titration. Also shown is hot water tank with resistance heater and magnetic drive water circulating pump. Also shown is separate low time constant continuous-flow temperature control system with shared controller.

**Catalytic Hydrolysis of Ethyl Acetate:** The apparatus consists of a jacketed glass reactor packed with Rohm and Haas Amberlyst ion exchange resin, which serves as an acid catalyst. A 20 liter polyethylene tank supplies an aqueous solution of ethyl acetate to a magnetic drive centrifugal pump, which feeds the acetate solution through a rotameter to a stainless heat exchanger and then to the reactor. A needle valve is used to set the flow rate, and reactor effluent samples are collected, timed, and titrated to a phenolphthalein end point using sodium hydroxide. A second centrifugal pump circulates hot water to the heat exchanger and the reactor jacket from
a resistance heated vessel. The water temperature is controlled by a commercial temperature indicator/controller.

The students set a volumetric feed rate and an operating temperature, allow a steady state to develop, and then analyze the reactor effluent to determine the conversion. From data taken at typically three feed rates and three temperatures, the students can calculate the activation energy and rate constant for the hydrolysis reaction.

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Power Consumption and Mixing Efficiency in Agitation

Model xxagt01

Agitation and mixing experiment, showing terminal box with ribbon cable to A/D board in computer, Servodyne stirrer motor controller on shelf, Cole-Parmer conductivity meter connected to conductivity probe in tank at bottom, variable speed DC stirrer motor on ball-bearing slide, fiberglass shaft with downward-driving impeller, 20 liter polycarbonate tank with tracer injection funnel, lamp and optical bead sensor.

Power Consumption and Mixing Efficiency in Agitation: The apparatus involves of a variable speed stirrer. The stirrer controller allows students to set the RPM in a range of 60 to 2000 RPM, and also to read the torque. Three polycarbonate tanks with removable baffles are supplied, as are a number of turbines and propellers of various sizes. The tanks can be mounted on a roller bearing based torque table equipped with a load cell connected to an Omega panel meter. Thus torque can be measured in two ways. The 20 liter tank is equipped with a conductivity cell mounted near the bottom and connected to a Cole-Parmer conductivity meter driving an RTD A/D board mounted in
a 486 or Pentium computer (computer supplied as an option). The tank is also equipped with an optical sensor and lamp assembly designed to detect the approach of small plastic beads suspended in the tank. Finally we supply a resistance heated aluminum cylinder equipped with a temperature sensor and driven by a variable transformer. This is used to determine the effect of stirred speed and impeller and baffle design on the heat transfer coefficient.

The experiment operates in four modes, namely:

- The students select a tank, impeller, baffle presence, and liquid (either water, Karo corn syrup, or catsup). Then the stirrer RPM is varied over a range, and the torque vs. RPM data are collected and plotted and compared to correlations in the literature. The data for a single torque vs. RPM run are obtained quite rapidly, but the many combinations of tank size, baffle presence, impeller design, and fluid type allow for extensive studies.

- Using water in the 20 liter tank, the students set an RPM and inject about 30 ml of salt solution using a funnel mounted at the top of the tank. As the tracer is dispersed, the transient conductivity at the bottom of the tank is digitized and recorded. Then a nonlinear regression program is used to determine two parameters of a six-pool model of the flow pattern in the tank. One of the parameters is the circulation rate in the tank, from which the mixing time can be calculated. Ten or more tracer injection runs can be made before refilling the tank.

- Several hundred plastic beads are added to the 20 liter tank, and the optical sensor is mounted on the side of the tank under the halogen lamp. A BASIC program acquires the sensor data, identifies bead entries into the illuminated region, and computes the entry interval distribution. This is typically a Poisson distribution, with characteristics that depend on stirrer speed.

- The aluminum heat transfer probe is mounted in the 20 liter tank, the probe power is set, and the temperature of the probe is determined as a function of stirrer speed. From these data the heat transfer coefficient can be calculated and compared to the literature.

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Membrane Air Separation
Model xxair01

Membrane air separation experiment, showing air filter and pressure regulator, two Permea membrane modules with valving to permit series or parallel operation, Omega pressure transducer with panel meter, needle valve for flow control, Sierra mass flow meters on tube side (low O₂) and shell side (high O₂) flows, Engineered Systems oxygen meters on tube and shell side streams. Air source is a standard cylinder of dry compressed air.

Membrane Air Separation: The apparatus consists of two Permea air separation modules, connected by stainless tubing and valves. Each module contains hundreds of polymeric tubes, the walls of which are more permeable to oxygen than to nitrogen. Dry air from a standard cylinder passes through a filter to a pressure regulator, which permits setting the operating pressure at which the modules operate. Air flow through the tube side (fiber lumen side) of the membrane modules can be parallel or series. Effluent air from tube sides of the modules is combined and passes to a needle valve used to control the
total tube side flow rate. It then flows to a Sierra mass flow meter and an Engineered Systems electrochemical.

The basic data for each run thus consists of the flow configuration (parallel or series), operating pressure, tube side flow rate and oxygen level, and shell side flow rate and oxygen level. A typical run takes about one minute, and consumes little air. The data can be processed to produce values for the oxygen and nitrogen permeability coefficients of the module fibers. The apparatus typically operates at room temperature.

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Photograph of the copper liquid-liquid extraction experiment, showing the copper and LIX + kerosine feed tanks, roller pumps with speed controllers, mixer-settler, variable speed stirrer, and raffinate and extract sample collection. The aqueous raffinate phase is analyzed for copper by a spectrophotometer.

**Copper Liquid-Liquid Extraction:** In this experiment one 20 liter polyethylene tank holds a copper sulfate solution at a level of five grams of copper per liter, and a second 20 liter tank holds a solution of LIX in kerosine. LIX is a mixed oxime that reversibly binds copper. Each tank feeds a variable speed roller pump, and the pump outputs are combined and sent to a small glass mixer vessel equipped with a variable speed stirrer. The mixer effluent passes to a settler which separates the aqueous and organic phases. The aqueous phase is analyzed for copper by a Spectronic 20 spectrophotometer.
The basic data for a run consists of the copper and LIX feed rates, the stirrer speed, and the copper level and the feed and aqueous effluent solutions. The data can be processed to determine the effect of feed rates and stirrer speed on the copper transfer rates. Depending on stirrer speed, copper removal can vary from 0 to 70 percent.

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Flash vaporization experiment, showing 20 liter feed tank with ball valve, magnetic drive centrifugal pump, flow control needle valve, glass vaporizer with internal 1000 W heater, glass flash vessel with overflow line, bottoms cooler, overheads condenser, bottoms and overheads sample flasks. Note also two temperature indicator/controllers and A1000 converter box, liquid and vapor thermocouples, liquid and vapor thermocouples, variable transformer for setting power level.

**Flash Vaporizer Dynamics and Control:** In this experiment water, or an ethanol/water mixture, is pumped into a glass vessel containing a 1000 W electrical heater driven by a variable transformer or by a commercial (Omega) PID controller. The heater effluent flows to a 150 ml glass flash vaporizer. Vapor from the vaporizer is condensed in a glass condenser, and the liquid bottoms flow to a glass cooler. Flow rates are measured by collecting and timing. With an ethanol feed the steady state behavior of the flash vaporizer can be studied. If the feed is water the apparatus becomes essentially a linear flow heater controlled by a PID controller. Step response runs can be made using the variable transformer to generate steps in heater power, or by stepwise changes in feed rate. The students can
set the PID gains and the controller set point, and observe sluggish control, good control, oscillatory decay of the error toward zero, or limit cycle oscillations depending on the PID gains. A parameter estimation program can be used to fit step response data.

Overhead and bottoms samples are most conveniently analyzed for ethanol using a gas chromatograph (optional).

Similar control studies can be done using an ethanol/water feed, but the system is no longer linear due to heat of vaporization and vapor/liquid equilibrium effects.

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Dye Mixer Dynamics and Control

Model mxr01

Dye mixer dynamics and control experiment, showing water pressure regulator, rotameter, dye solution feed tank, magnetic drive centrifugal pump, power strip with GFCI protector, terminal board for NI A/D board, stepper-motor driven needle valve, three flask flow system with ball valves, dye injection syringe, spectrophotometer with flow cuvette and connection to terminal board, stepper-motor control box, computer with National Instruments A/D board and LabVIEW software.

Dye mixer dynamics and control: In this experiment water flows from the mains through a rotameter and a needle valve into a flow system consisting of three 300 ml Erlenmeyer flasks. Ball valves allow for flow through three vessels in series, through one vessel only, or through a combination of vessels. The effluent passes through a special flow cuvette in a Spec 20 spectrophotometer, the signal from which is digitized and processed by a LabVIEW. A solution of methylene blue dye is held in a 20 L polyethylene carboy, flows to a
magnetic drive centrifugal pump, and then through a stepper-motor driven needle valve to mix with the water feed stream. The computer controls the dye solution valve via a stepper motor.

The first mode of operation involves using a syringe to inject a pulse of dye into the feed stream. The flow system response is digitized, plotted, and then the mean residence time (and thus the system volume) are calculated. The students can repeat the calculation using a spreadsheet, since the dye concentration vs. time data are written to a file. Each run takes only one or two minutes.

Next, a BASIC program is used to carry out an iterative nonlinear regression algorithm which estimates two parameters of a model of the flow system. Some of the water flows through three flasks and some through only one; the program is able to estimate the fraction passing through the three-flask portion, an otherwise unmeasurable quantity. This provides the students with a very simple and concrete introduction to modeling and parameter estimation.

In a second mode the water feed rate and flow system configuration are set, the dye feed pump is turned on, and the computer executes a PID control program. The students set the desired effluent dye concentration (the set point) and the algorithm adjusts the valve position to drive the effluent concentration to the set point, and thus the error to zero. The students are able to specify the three PID gains and the set point, and observe the nature of the control, which can range from sluggish to good to damped oscillatory to limit cycle oscillations. One nice result is to observe good control using a single flask only, and then see the system become unstable as two flasks are added to the flow path. This demonstrates clearly how additional delay in a loop can make control more difficult.

An important feature of the experiment is that the students can see directly all elements of a PID feedback control loop in action, including the passage of the dye through the flow system, the operation of the control valve, and the spectrophotometer response. They can also look at the statements in the program that implement the PID controller.

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Pump Characteristics Experiment
Model xxpmp01

Pump characteristics experiment, showing upper and lower polycarbonate tanks with connecting pipe and valve, inlet manifold, belt-driven gear pump and centrifugal pump, variable speed DC motor with torque arm and load cell. Flow circuit includes pump, pressure transducer, metering valve, orifice/differential pressure transducer, turbine meter, rotameter, return line to upper tank. Panel meters display pump RPM, discharge pressure, orifice differential pressure, and flow rate.

Pump Characteristics Experiment: In this experiment water flows from a 20 liter polycarbonate tank to a centrifugal pump or a gear pump. The pumps are belt-driven by a variable speed DC motor mounted on ball bearings. An arm on the motor bears on a load cell connected to a panel meter, and this allows the torque (and thus power) to be measured. A stainless pressure transducer driving a panel meter indicates the pump discharge pressure. Water discharged from the pump passes through an orifice/differential pressure cell combination, through a rotameter, and then through a turbine meter.
Water leaving the turbine meter flows to an upper 20 liter tank, and returns through a ball valve to the lower tank. Closing the ball valve allows an absolute measurement of flow rate by timing the rate a change of level in either tank. Students calculate the pump efficiency as a function of RPM from the torque/RPM and flow rate/discharge pressure data, and also calibrate the orifice, turbine meter, and rotameter over a range of flow rates. Space is available to add as options other flow meters such as vortex or ultrasound meters. Water can be replaced by a more viscous fluid such as a solution of corn syrup or glycerine, or a light oil.

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