

# SEQUENTIAL BATCH PROCESSING EXPERIMENT

## *For First-Year ChE Students*

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**B**atch and semicontinuous operations are often used within the chemical process industries. For example, the pharmaceutical, food, consumer products, and pulp and paper industries regularly use batch processing. Also, many chemicals are made on a semicontinuous basis. According to a recent AIChE survey of the entry-level job market, 60% of recent chemical engineering graduates are entering industries that use either batch or semicontinuous operations.<sup>[1]</sup> Yet the engineering curriculum devotes less than 10% of its instructional time to batch or semicontinuous operations. Further, if chemical engineers are making an impact in the area of industrial batch and semicontinuous processes, then it seems logical to introduce chemical engineering students to those operations early in their education.

### THE UNIVERSITY OF NOTTINGHAM

The University of Nottingham is located in the Midlands of Great Britain about 120 miles north of London. The total University enrollment is approximately 18,000 undergraduate and 3,500 graduate students. Chemical engineering is part of the School of Chemical, Environmental, and Mining Engineering. The School's enrollment is about 400 students, divided between courses in chemical, environmental, and

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\*\* Students arrive at Nottingham after a 2-year curriculum similar to US freshman and portions of a sophomore college-level curriculum. BEng and BS are similar curricula. MEng is similar to a MS Chem Eng.; however, the equivalent thesis component, listed as a research or design project, leans more toward application and advancement of current engineering practices.

mining engineering, undergraduate (3-year BEng and 4-year MEng\*\*), and postgraduate (taught MSc and research MPhil, PhD). In Great Britain, most university entrants have A-level, which is equivalent to two years of preparation beyond the high school degree.

As shown in Figure 1, the chemical engineering laboratory at the University of Nottingham is impressive even by United States' standards. The operating laboratory floor space is 128 by 32 feet (4,096 ft<sup>2</sup>) and has a 27-foot-high ceiling to accommodate tall equipment. There are five additional laboratories (approximately 1,000 ft<sup>2</sup> each) located on both sides of the main laboratory.

The University of Nottingham laboratory has a substantial support staff, with one chief technician in charge of four

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machinists, one electronics technician, and two full-time laboratory technicians who prepare experiments and watch students during lab operations. Two or three graduate students are also present during any particular laboratory day. Typically, ten to fifteen groups, composed of two or three students each, are in the laboratory on laboratory days (Tuesday and Thursday afternoons). Typical hours are from 2:00 to 4:30 P.M., but several experiments take longer.

The laboratory experience encompasses the first three years of Nottingham's chemical engineering course. Usually, experiments during a particular semester follow the chemical engineering lecture class schedule.<sup>[2]</sup> First- and second-year students spend approximately five sessions per semester in the laboratory, and the third-year students spend two sessions on

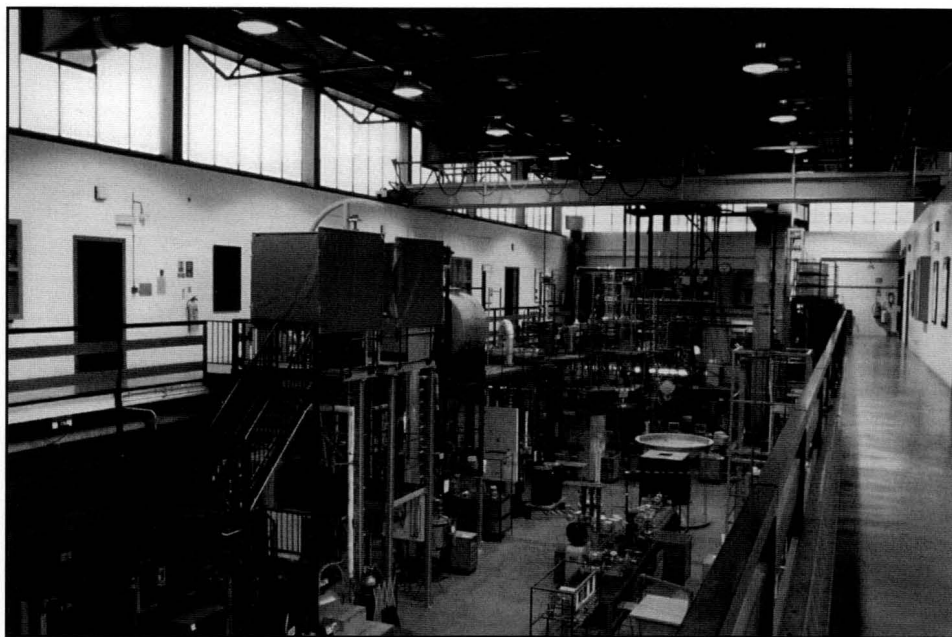


Figure 1. Nottingham's unit operations laboratory.

process control experiments. Those students who elect to take the BEng spend an additional five sessions working on a large unit operations experiment such as vacuum distillation, crystallization, liquid-liquid extraction, or filtration. These comprehensive experiments serve as term projects, and generally, one or two full-time faculty are present in the laboratory during a session. During the spring semester, the third-year MEng students are in the laboratory in place of the BEng for the term projects.

As mentioned above, experimental work is done continuously throughout the student's university career. The first-year students do simple experiments such as flow through pipes, flow through orifices, flow through pipe systems, velocity profiles, measurements of heat transfer coefficients, and mass/energy balances. These experiments are done concurrently while the students are taking lecture courses in fluid mechanics, heat transfer, and mass/energy balances. For example, the sequential controlled-batch plant experiment is performed by first-year students who are also taking the basic chemical-process principles course. The purpose of this brief summary is to allow the reader to make a rough comparison between U.S. and UK degree schemes. Grose provides a broader perspective on engineering education in the UK.<sup>[3]</sup>

#### FRESHMAN/FIRST-YEAR EXPERIENCE

There is renewed interest in introducing students to engineering concepts through hands-on experience. Many efforts, with good reason, are at bench scale. One example is the work

*... students  
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experience  
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equipment  
on a  
scale  
similar to  
that which  
they will  
encounter in  
industry.  
Observing  
and  
operating  
such a rig  
has no  
bench-scale  
substitute.*

of Hesketh,<sup>[4]</sup> which includes the reverse engineering of a coffee maker (conceived while a post doc in Great Britain). Another effort is the ongoing work by Perna and Hanesian<sup>[5]</sup> at the New Jersey Institute of Technology. They have taken several freshman engineering groups through a set of instrumentation/fluid-mechanics experiments with excellent success. But, again, these experiments are primarily bench scale.

The experiment described in this paper is on a larger scale. It is based around a 400-liter (about 100 gallons) vessel and uses steam at significant pressure (100 psig). Thus, students are able to experience process equipment on a scale similar to that which they will encounter in industry. Observing and operating such a rig has no bench-scale substitute.

## DESCRIPTION OF EXPERIMENT

The plant schematic is shown in Figure 2. The metering Tank M is filled with Feed A by Pump P and discharged into process Tank T via Valve A. High- and low-level sensors are provided for both Tanks M and T. Tank T is only partially filled by A, so Feed B is added until a Hi position is reached. At the same time, the agitator starts, and once full, the contents of the tank are heated by steam to 55°C and left for 10 minutes. The tank is then cooled to 40°C and the contents are discharged. Other sensors involved include those for temperature and valve position (open or shut).

The first step in the system design (discussed in more detail later) is to specify the sequence of actions required and then to identify the conditions necessary for a particular action. For example, Pump P operates if Tank M is low and Valve A is closed. To successfully manually operate or automate any batch process, it is very important to fully appreciate the process sequence logic—otherwise valves will be in the wrong position and pumps will be left on when they should be off.

Students were given two objectives for this experiment:

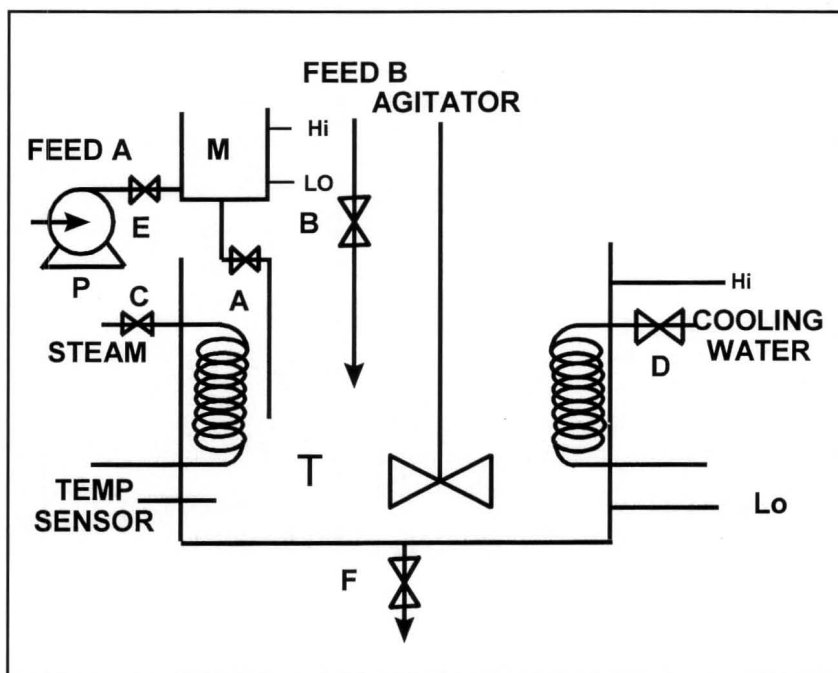


Figure 2. Sequential batch processing experiment schematic.

**TABLE 1**  
Process Sequence

1. Accurately measure quantity of expensive feed stock A charged to the reactor (which is initially empty) from the metering tank.
2. The agitator starts and the reactor is filled to a preset volume with cheaper reactant B. Meanwhile, the metering tank refills, ready for the next cycle.
3. Low-pressure steam is admitted to the jacket surrounding the reactor, and heating stops when the desired "high" temperature is reached.
4. Stirring is continued alone for a set reaction period.
5. Cooling water passes through a coil in the reactor and stops when the desired "low" temperature is reached.
6. Reactor empties and stirrer stops.
7. Cycle completed - ready to start at (1) again.

**TABLE 2**  
Operating Check-List

1. \_\_\_ Tank M at Hi level (high-level panel light is on)
2. \_\_\_ Tank T at Lo level (low-level panel light is on)
3. \_\_\_ Valve A is closed (panel light is off)
4.  Mains water (i.e., B) supply is available
5. \_\_\_ Valve B is closed (panel light is off)
6.  Steam supply available
7. \_\_\_ Valve C is closed (panel light is off)
8.  Cooling water supply available
9. \_\_\_ Valve D is closed (panel light is off)
10. \_\_\_ Pump P is off (no sound)
11. \_\_\_ Valve E is closed (panel light is off)
12. \_\_\_ Valve F is closed (panel light is off)
13.  Lower supply reservoir filled with A and ready

Signature of Group Member Verifying the Check

*If any of these conditions are not correct, notify Prof. Willey or a technician before continuing.*

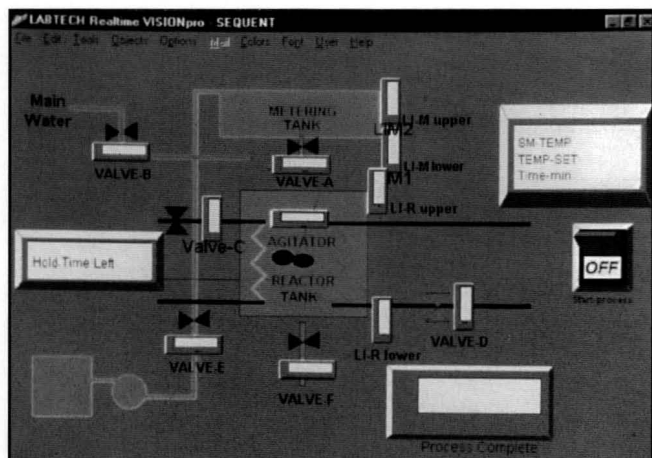
- To obtain experience with a sequentially controlled batch plant and to gain appreciation of the advantages resulting from automated operation.
- To perform heat balances on the heating and cooling operations that form part of the batch cycle.

To help students understand the process sequence, they are provided with Table 1, which can be read while they follow the automated operation on the control panel. To reinforce the importance of having all components in the

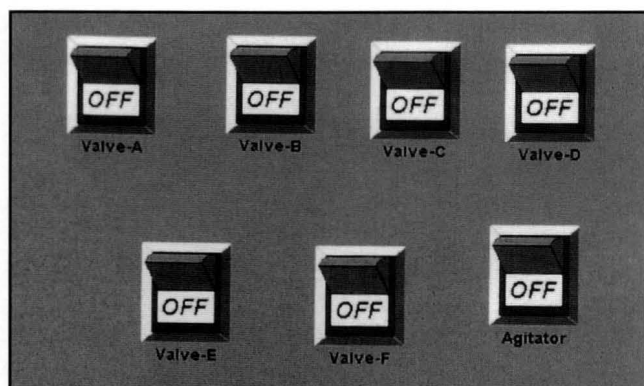
correct mode before starting a batch, they also receive a Check-List (see Table 2).

### IMPLEMENTATION

This sequence is implemented in two ways. First, the experiment is done under automatic control; the students simply initiate the experiment by moving the on-off switch located on a computer monitor (see Figure 3) to “on,” using a mouse. In the second experiment, students implement the sequence and record data manually. They use thermometers, stop watches, and toggle switches located on the computer monitor, as shown in Figure 4.



**Figure 3.** LabTech Vision™ screen created for automatic control. A mouse pointer is used to initiate the On/Off switch.



**Figure 4.** LabTech Vision™ screen created for manual control. Students use a mouse pointer to initiate the various On/Off switches in correct sequence.

**TABLE 3**  
Summary of Steps Involved in Sequential Batch Experiments

Step No.	Operation	Proceed on Condition That...	Initiated by	Ended by	Action Necessary
1	• Fill Tank M with Feed A	• Valve A closed	• Tank M low	• Tank M high	• Open Valve E • Start Pump P
2	• Discharge Tank M to Tank T	• Tank M high • Valve F closed • Valve E closed • Tank T low • Pump P off	• Start: Push Button	• Tank M low	• Open Valve A
3	• Add Feed B • Start agitator		• Tank M discharged into T	• Tank T high	• Open Valve E • Close Valve A • Start agitator
4	• Heat to 55°C		• Tank T high	• Temp at 55°C	• Open Valve C • Close Valve B
5	• Wait for the duration specified		• Temp 55°C	• Timer	• Start Timer • Close Valve C
6	• Cool to 40°C		• Timer	• Temp at 40°C	• Open Valve D
7	• Discharge	• Valve A closed • Valve B closed	• Temp at 40°C	• Tank T low	• Open Valve F • Close Valve D
8	• Process Complete		• Tank T low	• Start of new batch	• Light warning lamp • Stop agitator • Close Valve F

### Programming Required to Develop the Experiment

The biggest challenge is programming LabTech Control<sup>[6]</sup> to operate a batch process. It has the capability to read analog signals, to record data to diskette, and to display data on computer monitors. It also has the ability to perform PID and on-off control.

The analog signals acquired are four level sensors, 0.2 volts when low and 2.5 volts when high. Signals are also acquired from thermocouples reading the batch temperature and the cooling water temperature. The signals sent out are all digital (Hi or Lo). They control the agitator (on or off), the opening of Valve A (which drains Tank M), Valve B (which controls the admission of Feed B to Tank T), Valve C (which controls the admission of steam for heating), Valve D (which controls the admission of cooling water), Valve E (which controls the refilling of Tank A), and Valve F (which controls the draining of Tank T). These valves have to be turned on in the correct sequence for the batch experiment to operate correctly. In LabTech this is done by using two stages triggered by the proper conditions. For example, Stage 1 for Valve A is a 1-Hz stage of 1 second. It is triggered open when the students switch the on-off switch (located on the computer monitor) to "on" by using the mouse. Stage 2 for Valve A is a 1-Hz stage of 7200 seconds (the experiment takes about 3600 seconds) triggered on after the Lo-Level sensor in Tank M indicates that Tank M is empty. When Tank M is sensed empty, Valve A closes and stays closed for the duration of the experiment. Valve B is triggered open (Stage 1) by the same Lo-Level sensor. It is triggered closed (Stage 2) when the water reaches the Hi-Level sensor in Tank T. Table 3 gives the sequence of events programmed through LabTech Control using essentially two stages—one to initiate and one to terminate the desired action.

Desired batch temperatures are read from a data file that is set up beforehand which contain the desired batch temperatures. In this case, the initial set temperature is 20°C (ambient) (a LabTech Stage immediately reading at frequency of 1 Hz and on for only

0.5 second, thus only one point is read). When Tank T becomes full, the high temperature (55°C) setpoint is read when triggered by a Hi signal received from the Hi-level sensor located in Tank T. After the tank reaches this temperature, another LabTech Stage with a frequency of 1 Hz over the hold time (in Hz) is used to read the same temperature (55°C), followed by the lower cooler dump temperature (40°C).

### RESULTS ACQUIRED BY STUDENTS

For the first run, data are acquired to ASCII data files by using LabTech Control data acquisition capabilities. Information recorded is: time of sampling event, temperature of Tank T contents, and the outlet temperature of the cooling water. These are acquired at a frequency of 0.0167 Hz (or every minute). For the second run, data are acquired manually by recording temperature every minute into a laboratory notebook. In groups that comprise three students, one student controls the sequence of the experiment while another

Event	AUTOMATED CONTROL (mins)	MANUAL CONTROL (mins)
Valve A Opens	0.15	0.00
Valve A Closes, Valve B Opens + Mixer	1.25	1.08
Valve B Closes, Valve C & E Opens	2.67	2.28
Valve E Closes	3.92	3.92
Temp. reached set point 55 C	13.13	13.33
End of Holding Time (10 Mins), Valve D Opens	23.15	23.37
Valve D Closes, Valve F Opens	32.90	37.50
Valve F Closes	42.55	46.50
END	42.57	46.50

Figure 5. Event times reported by a student for the two experiments: automatic and manual control.

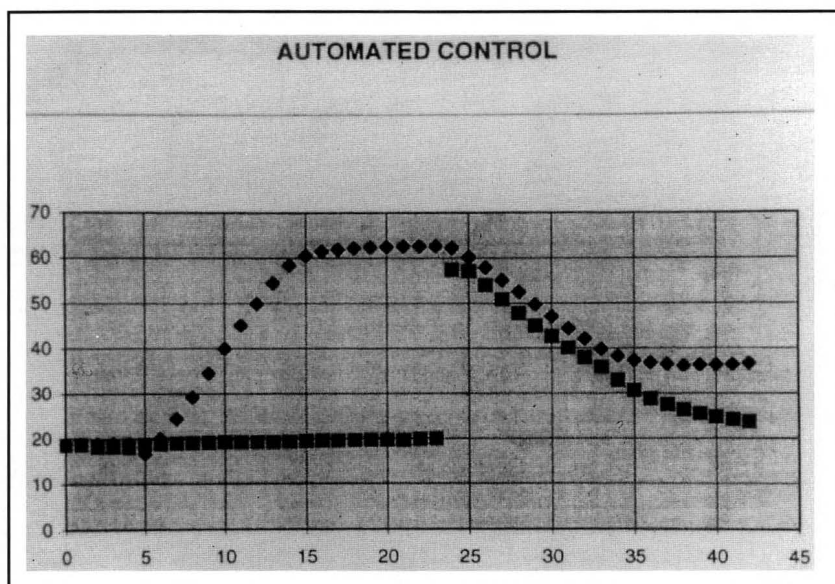


Figure 6. Temperatures, °C, acquired by Lab Tech as a function of elapsed times, mins, as presented in a student's report.

student records temperature data, and the third student collects steam condensate, calibrates the rotameter, and monitors cooling water flow through the rotameter.

Figures 5 and 6 are results taken from a student report. Figure 5 shows timed events for the two methods of operation. In this case, the student noted in his report that the automatic control was faster (by about 10%) and therefore manual control is less efficient, and that over time this would equate to a 10% decrease in production rate.

Figure 6 shows a very smooth temperature profile acquired by LabTech Control and later plotted by the student using Microsoft Excel. Series 1 represents the batch temperatures (in Tank T), and Series 2 represents the cooling water outlet temperature. We see that it took about ten minutes to heat the batch to 62°C (set point was 55°C—on/off control was used in this case) and that the batch held at this temperature for the required ten minutes. Cooling followed.

It is interesting to see how close the cooling water exit temperature approached the batch temperature. These first-year students had not had a course in heat transfer at this stage, so they did not recognize how efficient the cooling coil inside the vessel performed. They did note that the plots of temperature acquired automatically were smoother compared to temperatures acquired manually. Figure 7 shows a set of data collected manually as read from thermometers by students. The “noise” observed in this particular figure is comparably low for manually acquired data typically collected by students.

Students also made observations about automatic-versus-manual control. One student noted that when the plant is operated under automatic control, operators are free to do other vital jobs. He also noted that running the rig remotely (over the Internet!) would maintain a safe distance for dangerous reactions.

The students were also required to do energy balances around the reactor for both the heating and cooling operations. For the heating cycle, students typically reported a heating efficiency of about 45% (calculated as [Heat

absorbed by water]/[Heat released by condensing steam]). Two explanations exist. One, that the apparent steam condensate collected is large because it included hold-up from previously condensed steam; the second explanation, which most students mentioned, was that the stainless reactor itself had thermal capacity and also required heating. Heat losses also exists, but are relatively small in comparison.

What did we discover during the first few runs? We missed telling the students to record the inlet cooling water temperature; this has now been included in the procedures. Hind-sight is 20/20.

## CONCLUSIONS

The experiment provides a worthwhile educational experience for relatively inexperienced students. In particular, the advantages of automated operation are demonstrated. Further, students are able to practice their IT skills and to apply basic energy-balance techniques.

## ACKNOWLEDGMENT

The authors acknowledge Thomas Holgate and Tracy Wong, first-year students at the University of Nottingham, whose data were used as examples in this paper. The authors also acknowledge the assistance of Fred Anderton in wiring the circuits required to set up the automation of the sequential control unit. Prof. Willey acknowledges Northeastern University for permission to do a sabbatical at the University of Nottingham during the fall of 1997.

## REFERENCES

1. Graham, E. Earl, *AICHE Extra*, 4, Sept (1998)
2. Jones, W.E., “Basic Chemical Engineering Experiments,” *Chem. Eng. Ed.*, **27**, 52 (1993)
3. Grose, T.K., “A Yankee Engineer in Queen Elizabeth’s Court,” *ASCE Prism*, 28, March (1999)
4. Hesketh, R.P., “Wake-Up to Engineering!” *Chem. Eng. Ed.*, **30**, 210 (1996), and R.P. Hesketh and C.S. Slater, “Hands-On Freshman Engineering at Rowan University,” *AICHE 1998 National Meeting, Session 175—The Freshman Experience*; November (1998)
5. Perna, A.J., and D. Hanesian, “The NJIT Freshman Experience: A Historical Perspective,” *AICHE 1998 National Meeting, Session 175—The Freshman Experience*; November (1998)
6. LabTech User’s Guide, Laboratory Technologies Corporation, 400 Research Drive, Andover, MA (1999) □

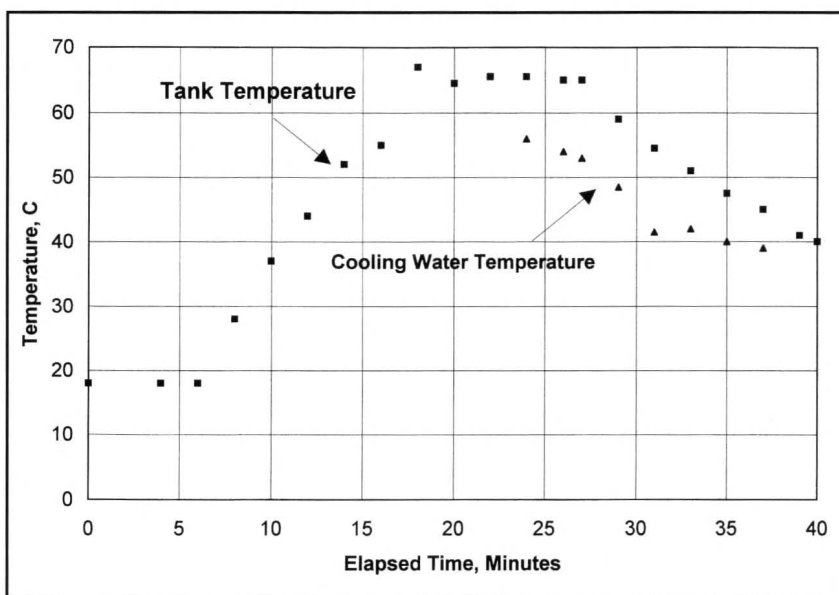


Figure 7. Temperatures as a function of elapsed time as recorded by a student group during the manual run.