ChE laboratory

A FIRST CHEMICAL ENGINEERING LAB EXPERIENCE

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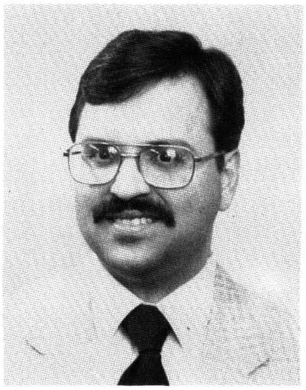
This paper describes a simple thermodynamics experiment currently in use at Villanova University. The specific heat of a liquid is determined using an easily and inexpensively constructed, operated, and maintained apparatus.

ONE OF THE major challenges for faculty involved in a first chemical engineering lab course which emphasizes technical report writing is the development of appropriate experiments. Because the students have limited chemical engineering experience at the start of such a course, the first few experiments must be simple and should be based on chemical engineering courses completed in the sophomore year.

This paper describes a simple thermodynamics experiment currently in use at Villanova University. The specific heat of a liquid is determined using an easily and inexpensively constructed, operated, and maintained apparatus. The students use the results of the experiments to formulate recommendations on how the performance of the apparatus can be improved so that it could be used in a wider range of applications than those specifically tested through experimentation.



Vito L. Punzi joined the chemical engineering faculty at Villanova University in 1980 after previous experience in industry. He received his BS degree from Polytechnic Institute of Brooklyn in 1972 and his MS and PhD from Polytechnic Institute of New York in 1974 and 1979, respectively. His teaching interests are in chemical process calculations, chemical engineering thermodynamics, and fluid dynamics, and he is currently performing research on the solute rejection mechanism in reverse osmosis and on the removal of heavy metals from industrial waste water via adsorption.

Since the experiment produces accurate results and stimulates the development of "engineering judgment," a satisfying technical report writing experience usually results.

APPARATUS

The principal components used in this experiment are shown in Figure 1. A wide-mouthed 700 ml Dewar flask vacuum bottle is fitted with a styrofoam insulated lid which supports a 300 watt immersion heater and a mercury-in-glass thermometer. The flask is mounted on a magnetic stirrer to ensure proper mixing and a uniform liquid temperature. The immersion heater is connected to a variable power transformer and wattmeter (accurate to ±5%) so that the energy input rate to the liquid can be controlled and determined. A 500 ml graduated cylinder and hydrometers are used for the volume and specific gravity measurements needed to determine the mass of the liquid samples. A digital stopwatch is used along with the thermometer to collect elapsed time and liquid temperature data.

THEORY

The unsteady state energy balance for a nonflow process with negligible kinetic and potential energy effects can be expressed as

$$Q_{net} - W_{net} = [dU/dt] \qquad (1)$$

where Q_{net} is the net rate of heat transfer into the system from the surroundings, W_{net} is the net rate of work performed by the system on the surroundings, and U is the internal energy of the system, which varies with time, t. If the system undergoes changes at constant pressure and if shaft work effects are negligible, the thermodynamic definitions of work and enthalpy (H) can be used to obtain

$$Q_{net} = [dH/dt] \qquad (2)$$

For the calorimeter used in the experiment (a Dewar flask vacuum bottle), the thermodynamic system is the liquid contents of the calorimeter, the solid components of the immersion heater and the inner wall of the Dewar flask. In this system, Q_{net} is the sum of the heat added to the system through the immersion heater (Q), and the heat added to the system through the inner wall of the Dewar flask due to the temperature difference between the surrounding environment and the system. Since the heat added to the system through the inner wall of the Dewar flask is negligible when compared to the heat added to the system through the immersion heater, Eq. 2 simplifies to

$$Q = c_p [dT/dt] \qquad (3)$$

where c_p is the total heat capacity of the system considered, and T is the temperature of the system, assumed uniform throughout the system.

The total heat capacity of the system consists of the heat capacity of the liquid contents, c_{pl}, and the heat capacity of the solid components of the immersion heater and the inner wall of the Dewar flask which are lumped into a single parameter, the calorimeter constant c_{pc}. If the heat capacity of the liquid is expressed in terms of the specific heat of the liquid (c_{pl}') and the mass of the liquid (m_l), then Eq. 3 can be rewritten as

$$Q = (m_l c_{pl}' + c_{pc})[dT/dt] \qquad (4)$$

Thus, a well-insulated batch calorimeter can be used to determine the specific heat of a liquid from the rate of temperature change which results when energy is added at a known rate to a known liquid mass.

EXPERIMENTAL PROCEDURE

First, the students perform three repetitions of a calibration experiment to determine the calorimeter constant c_{pc}. A constant energy input rate between 100 and 200 watts is used to heat 500 ml of water. Since the specific heat of water is known and is relatively constant, rate of temperature change data can be used to determine c_{pc} using the following rearrangement of Eq. 4

$$c_{pc} = (Q/[dT/dt]) - m_l c_{pl}' \qquad (5)$$

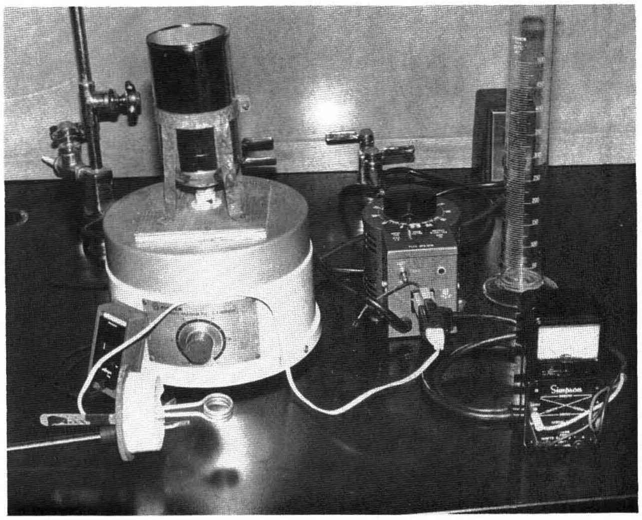


FIGURE 1. Experimental Apparatus

Next, a series of experimental runs is performed to determine the specific heat of the test solution, c_{pl}', at several concentrations. The same procedure is used as in the calibration experiments, except that a test solution is used instead of water. Using the value of c_{pc} determined above, the value of c_{pl}' can be calculated from

$$c_{pl}' = \{(Q/[dT/dt]) - c_{pc}\}/m_l \qquad (6)$$

Ethanol-water solutions and glycerol-water solutions have been used successfully as test solutions. These solutions have been used primarily because minimal evaporative losses occur, and because extensive specific heat data are available in the literature.

The test solutions are heated over a narrow temperature range chosen so that the middle of the range coincides with a temperature at which literature values of the specific heat are available. For example, glycerol-water solutions are usually heated from 5°C to 25°C, since literature data are available at 15°C (1). If calibration runs are performed over the same temperature range, the final results of the experiment are usually quite accurate.

The experiment can be performed either using standard solutions of known composition or using solution compositions chosen at random by the students. The advantage of the former approach is that it allows a direct comparison between experimental and literature values.

RESULTS

The heat capacity of the solid components of the calorimeter (c_{pc}) is approximately 10% of the total heat capacity of the system. Thus, neglecting this parameter in an energy balance would result in a significant deterioration in the quality of the final results. However, the magnitude of this parameter is small enough so that more accurate estimates would not significantly improve the final results.

During the 1984-85 academic year, ethanol-water solutions were used, with solution compositions chosen at random by the students. All experimentally determined values of specific heat were within 10% of an interpolated literature value, at all concentrations.

During the 1985-86 academic year, standard glycerol-water solutions containing 10 wt% glycerol and 40 wt% glycerol were used; a limited number of additional experiments were performed using a 20 wt% glycerol solution. As shown in Table 1, the experimentally determined values of the specific heat of the 10 wt% solution were, on the average, within 2% of the literature value at 15°C. All but one of the 38 individual values determined in the experiments using the 10 wt% solution were within 7.5% of the literature value at 15°C, with 32 of the 38 individual values within 5% of the literature value at 15°C.

TABLE 1
Results of Experiments Using Glycerol-Water Solutions at 15°C

Composition (weight % glycerol)	Specific Heat, cal/g-°C			Number of Samples
	Range of Individual Values	Average Experimental Value	Literature Value(1)	
10	0.866-1.002	0.941	0.961	38
20	0.859-0.893	0.881	0.929	3
40	0.752-0.849	0.791	0.851	37

The experimentally determined values of the specific heat of the 40 wt% solution were, on the average, within 7% of the literature value at 15°C. All of the individual values determined in the experiments using the 40 wt% solution were within 11.5% of the literature value at 15°C, with 33 of the 37 individual values within 10% of the literature value at 15°C.

DISCUSSION

The exercise described above is structured so that the student is required to do more than collect data and tabulate results. For many students, this experiment provides them with their first opportunity to develop "engineering judgment" in the chemical engineering laboratory environment. The students must use knowledge obtained in lecture courses, and the results of experimentation, to make logical conclusions and meaningful recommendations. Further, the students are required to organize and present their findings in a technical report written in accordance with the classroom instruction they are given.

This . . . experiment involves basic thermodynamics, uses easily operated experimental equipments, produces accurate final results . . . and encourages students to use engineering judgment . . .

The experiment is not intended to produce handbook-quality physical property data, nor to duplicate industrial research methods. Indeed, more accurate results could easily be obtained, for example, by using a more accurate wattmeter or by using a direct measurement of sample mass. However, despite these and a few other built-in flaws in the equipment and procedures used, reproducible results which are within ±10% of handbook data are consistently achieved. The flaws do not significantly deteriorate the quality of the results produced by the apparatus, but are obvious enough so that the students can usually make several worthwhile recommendations, rather than the contrived recommendations which are quite prevalent in undergraduate reports.

CONCLUSIONS

This paper describes an experiment which involves basic thermodynamics, uses easily operated experimental equipment, produces accurate final results from a rather straightforward analysis and encourages students to use engineering judgment to formulate worthwhile conclusions and recommendations on how to improve the apparatus. All of these features combine to produce a favorable first experience in technical report writing.

In addition, the experiment requires only a small expenditure of funds: since breakable parts are relatively inexpensive, annual operating costs are usually minimal and most of the equipment involved in the experiment need not be committed solely to this experiment since it can be used in other teaching, laboratory, and research applications.

ACKNOWLEDGEMENTS

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REFERENCE

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