A Smallscale Glass Calorimeter

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Abstract: A smallscale glass calorimeter has been designed and constructed to reduce around 90% of the volume of reagents required for thermochemical experiments performed in Dewar flasks and Styrofoam or polystyrene cups. The description and calibration of the calorimeter is shown and one example is given in order to illustrate the advantages of working with a transparent calorimeter.

Introduction

A mini glass calorimeter (Figure 1), which has been designed to reduce the volume of reagents required for thermochemical experiments, is described. It was constructed as follows: Two flat-bottom glass tubes 2 × 9 cm (interior) and 5.5 × 11 cm (exterior) were sealed joining their tops under the flame. These dimensions allow an inside maximum volume of 27 mL.

Before the construction of this calorimeter, a 500-mL plastic-coated calorimeter was used in our student laboratory in which the minimum volume employed was 200 mL. Now we use only 9 to 20 mL of reagents depending on the experiment. The benefits of working on a small scale [1] include reducing chemical use, thus waste disposal; shortening experimental time; and saving storage space. Moreover, when solution experiments were conducted in the plastic-coated flask, into which we could not see, we often noticed that some salt remained undissolved at the end of the experiment. That is also a problem with Styrofoam or polystyrene cups used as calorimeters. The transparency of this mini calorimeter adds an extra advantage: when dissolving a salt we observe that the temperature increases (exothermic reactions) or decreases (endothermic reactions) until the salt is completely dissolved, then the temperature remains constant and this temperature can be recorded precisely as the equilibrium temperature.

The main requirement of a calorimeter is that it behaves adiabatically during the measurements. For this reason, the glass device has been designed with an insulating air chamber that takes advantage of the low thermal conductivity of air. To increase isolation from the surroundings, a vacuum can be achieved through a picked hole in the outer glass cylinder.

Calibration of the Calorimeter

10 mL of room temperature water are placed inside the calorimeter and a mini stir bar is added. The calorimeter is placed on a magnetic stir plate and the stir function is turned on. The calorimeter is closed using a rubber cap into which a thermometer is inserted and the temperature is recorded. (The thermometer we use is digital with an accuracy of 0.1 ºC.)

10 mL of hot water is obtained, its temperature recorded, and immediately added to the room temperature water in the calorimeter. The calorimeter is again closed with the rubber cap and the temperature is recorded every 20 s for 5 min. Time (*x* axis) is plotted versus temperature (*y* axis). See Figure 2.

A straight line is drawn through the points and extrapolated to obtain the *y* intercept. This will be the equilibrium temperature (t_e) in the following equation:

$$-C_w\, m_{hw}\,(t_e - t_{hw}) = C_w\, m_{cw}\,(t_e - t_{cw}) + K(t_e - t_{cw}) \qquad (1)$$

where m_{hw} is the mass of the hot water, m_{cw} is the mass of the room temperature water, C_w is the specific heat of water (4.18 J g^{-1} K^{-1}), and K is the heat capacity of the calorimeter or calorimeter constant, the only unknown quantity in eq 1. This procedure allows calculation of the calorimeter's heat capacity.

Example Experiment: Determination of the Heat of Solution of KNO_3

This calorimeter is suitable for the experiments given in references 2 and 3 as well as that outlined below.

9.0 mL of room temperature water is placed inside the mini calorimeter. A stir bar is added and the magnetic stir plate turned on. The calorimeter is closed with the rubber cap, which has the thermometer inserted, and the temperature, t_w, is recorded. 1.0 g of KNO_3 is added to the stirred water inside the calorimeter. The calorimeter is closed and the lowest temperature, t_e, is recorded. The heat of the reaction can be calculated as

$$Q_{sol} = C_w m_s(t_e - t_w) + K(t_e - t_w) \qquad (2)$$

where m_s is the mass of the water plus the mass of salt (10.0 g), C_w is the specific heat of water (4.18 J g^{-1} K^{-1}), and K is the heat capacity of the calorimeter calculated using eq 1. From eq 2 and the moles of KNO_3 (n), the enthalpy of solution, ΔH^0_{sol}, is $-Q_{sol}/n$. The calculated heat of solution for our experiment was 35.7 kJ mol^{-1}. The experimental result is in very good agreement with the literature value, 34.89 kJ mol^{-1} at 25 °C [4].

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Figure 1. Photograph of the calorimeter containing 20 mL of a colored solution for the sake of viewing.

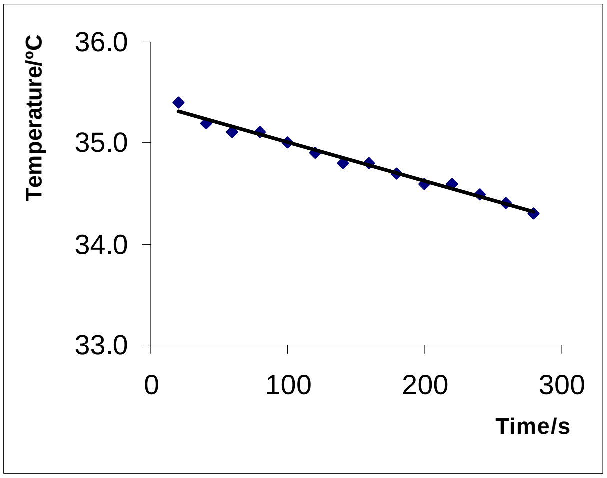

Figure 2. Temperature versus time plot obtained during calibration of the calorimeter.

References and Notes

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