

The two reactions measured in this way are report

[Technology](#), [Development](#)

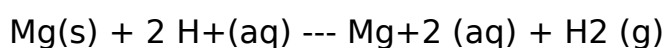


Introduction

In this experiment, we are interested to determine the heat change of two different reactions using the ice calorimeter. The heat change values are applied together with the given values of the heat transformation of water ($\Delta H(\text{H}_2\text{O})$) and the application of Hess law to establish the heat of formation of MgO ($\Delta H(\text{MgO})$)

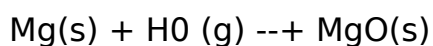
A formation reaction has a defined stoichiometry which makes them ideal constructs to be investigated by the Bunsen ice calorimeter. When the heat is released by the reaction, it causes the ice to melt and the volume of the ice-water mixture to contract. From the volume contracted, the amount of ice melted can be calculated. The reaction takes place in a test tube surrounded by an ice-water mixture of defined volume.

Heat released by the reaction leads to the melting of the ice and a contraction in volume of the ice-water mixture. At constant pressure (the test tube is open to the atmosphere) the heat of reaction $q_p = \Delta H$. From the observed volume contraction, the amount of ice melted can be calculated and combining this with the known value for $\Delta H(\text{H}_2\text{O})$ gives the heat (J) produced by the reactants.



A value for the enthalpy of formation of water is available from the literature.

These three reactions can be combined to yield the formation reaction of magnesium oxide.



The enthalpy state functions of equations (1&2) are measured using the procedure below equation 3 is the formation reaction of H₂O (f).

THEORY

The ice calorimeter is a simple robust apparatus in which 5% precision is easily reached in the measurement of enthalpy changes in fast exothermic reactions. The apparatus is depicted below. The calorimeter is constructed from a 250 mL electrolytic beaker, a test tube, a 1 mL pipette graduated to 0.01 mL, a rubber stopper, and short pieces of 5 mm glass tubing. It is important to have the test tube in the center of the electrolytic beaker with the bottom of the test tube well removed from the base of it. The diameter of the glass rod and rubber tubing should be chosen such that the rod is held firmly by the tubing, but at the same time may be slid up and down to act as a plunger.

Figure 1. Schematic View of the Bunsen Ice Calorimeter

Figures 2&3. View of the Bunsen Ice Calorimeter

The electrolytic beaker is filled to the top with a mixture of ice and water and the stopper assembly put firmly into place with the glass rod plunger removed from the rubber tubing. It is helpful to cool the rubber stopper to ice temperature before inserting it into the beaker. Care should be taken

that the tip of the test tube is surrounded by ice particles. It is critically important that the approximately 1: 1 water-ice mixture is stirred to remove as many air bubbles as possible.

The entire assembly is placed in a plastic tray and covered with ice. Ice is piled high over the top of the stopper and as high as the lip of the test tube. The acid reactant is transferred into the test tube via a pipette and allowed to come to thermal equilibrium. The solid Mg metal or MgO can be cooled to 0 °C but it should remain dry. Water is introduced down the rubber tubing until full; the plunger is then placed in the rubber tube and pushed in so that water rises as high as possible in the pipette.

The rate of melting of ice in the electrolytic beaker is followed by observing the rate of fall of the level in the pipette; since the density of water is greater than that of ice, the volume contracts as the ice melts. After monitoring this contraction for ca 5 minutes, the second reactant is added to the test-tube. The reaction ensues and the heat generated is transferred to the ice jacket in the electrolytic beaker at 0°C, where it melts part of the ice and causes the pipette level to fall rapidly. When the reaction is over, the rate of fall of the pipette level returns to its original steady value. Throughout the procedure, pipette level is followed as a function of time, a reading being taken every thirty seconds or some other suitable interval. The results can then be plotted to determine the volume change due to the melting of the ice caused by the reaction.

Procedure

1. Loosely fill the calorimeter beaker with ice up to the level of the rubber stopper assembly.

2. Add iced water up to the same level with stirring to remove all air bubbles.
3. Place the stopper assembly on the beaker and fit the upper and lower retaining rings. Engage the three locking screws evenly making sure you do not over tighten them.
4. Gradually add iced water (Pasteur pipette) through the flexible entry tube until the water level rises in the graduated measuring pipette.
5. When the water level is about $\frac{1}{2}$ up the graduated pipette, insert a glass rod into the flexible tube so that the water level rises almost to the top. Do not push the rod in too far as it will be difficult to remove.
6. Place the assembled calorimeter in the plastic tray and cover with ice.
7. Place an accurately measured aliquot of the appropriate acid (5.00 mL) in the calorimeter reaction tube and leave the system to achieve thermal steady state.

Make Sure: You use the correct acid concentration and make a note of the actual concentration of each acid used. As the acid is the limiting reagent, accuracy is vital.

8. While the equilibration is taking place, use the reaction stoichiometry to determine the correct amount of magnesium or the oxide needed. Measure out an amount 10% in excess of the stoichiometric equivalent.
9. 'Zero' the calorimeter and commence readings of the change (decrease) in pipette volume at 30 second intervals.
10. Once V/t is reasonably steady, add the (metal or oxide) reactant and continue taking readings as the reaction proceeds. In order to ensure the

reaction goes to completion, use a

Pasteur pipette to wash down any MgO that might have adhered to the side of the reaction test tube using the acid in the test tube. Continue taking readings until the rate of change again returns to the value observed before the reaction started. Make sufficient additional readings to complete the extrapolation (see Fig. 4).

11. Repeat steps 1 to 10 using the other reagents.

Interpreting Observations

Since the ice calorimeter is operating at a temperature lower than ambient, the ice inside it is constantly melting. Covering the calorimeter with ice slows this spontaneous process but achieving a stable level in the pipette is not possible. Your data from each trial will appear somewhat like figure 4 below. Data taken at 0.5 minute intervals before addition of the reactants will show a gradual melting of ice and decrease in total volume. Likewise, after the completion of the reaction there will still be a gradual melting of the ice and thus determining the change in volume due to the reaction requires some interpretation.

Figure 4. Expected changes in the pipette volume during an experiment in the ice-calorimeter.

Because of the outer packing of ice/water and the excess of ice in the electrolytic beaker, the melting of the ice is carried out at 0 C. Under these conditions, the relation between the volume change (ΔV) and the weight of ice melted is derived as follows:

$$\begin{aligned}\Delta V &= V_i - V_w \\ &= w_i \rho_i - w_w \rho_w\end{aligned}$$

$$w = \Delta V \rho_w \rho_w - \rho_i$$

where V_i and V were the volumes of the ice melted and water formed during the reaction, W is the weight of the ice melted, and ρ_i and ρ_w stand for the densities of ice and water respectively at 0 °C. The heat evolved in the experiment (q_p) is given by the expression:

$$q_p = \Delta V \rho_w \rho_w - \rho_i \text{ joules}$$

where ΔV is in cm^3 and L (J g^{-1}) is the latent heat of fusion of ice. $\Delta_f H$ is thus given by q_p/m

where m is the number of moles of limiting reactant used in the test tube.

$$q_p = \Delta V \rho_w \rho_w - \rho_i \text{ joules} = -0.6250 \cdot 9167 \times 0.99980 \cdot 9998 - 0.9167334 \cdot 6$$

joules

$$= -5904.5 \text{ J}$$

$$\Delta H = q_p/m = -5904.5 \text{ J} / 18 = -328. \text{ kJ mol}^{-1}$$

$$\Delta_f H_{\text{MgO}} = \Delta_f H_{\text{Mg}^{2+}} + \Delta_f H_{\text{H}_2\text{O}} - \Delta_f H_{\text{MgO}} = -328 - 77 + (-286) = -537 \text{ kJ mol}^{-1}$$

Results and discussions

Both reactions of magnesium oxide and magnesium had a percentage error of around 55% and the comparison with the literature value indicates that just 55% of the substances reacted. One possible cause of this is the lack of reactants in the reacting solution. Other cause may be loss of heat from the reaction.

Conclusions

Bibliography

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