Introduction

The kinetics of base catalysed ester hydrolysis has been used as a test reaction for the comparison of isothermal calorimetry systems. In most calorimeters this is a lengthy process as in order to get an Arrhenius plot to derive the activation energy the process needs to be conducted at several temperatures. The Micro Reaction Calorimeter allows rapid changes in temperature saving days in experimental time over traditional calorimetry measurements. The hydrolysis of MethylParaben is already reasonably well understood and if the experiment is conducted under the conditions used here the system will follow pseudo 1st order kinetics.

Experimental

0.381g of MethylParaben (methyl 4-hydroxy benzoate) was dissolved in 50ml of NaOH with vigorous stirring. 1ml of this solution is then placed in the sample cell (1ml of the sodium hydroxide solution was placed in a reference cell). The cell was then placed in the Micro Reaction Calorimeter which was set to 15°C.

The system was allowed to stabilise before the temperature was then adjusted stepwise to 20°C and finally 25°C (allowing sufficient time between temperature changes for stabilisation).

Results

The step temperature change experiments produced the data shown below. The analysis of this data (shown below) shows the how the rate changes with temperature. Using this data to produce a kinetic Arrhenius plot gives a very good linear correlation. From this data an activation energy of 58 kJ.mol⁻¹ was calculated. The activation energy from this experiment compares very well with published data (60±2 kJ.mol⁻¹ and is well within the error limits for this type of experiment.
Discussion and Conclusions

As well as examining the kinetics of this process it is important to measure the thermodynamics of the system. Three other experiments were conducted to verify the heat of reaction all of these fell within the literature value where $\Delta H = -50 \pm 2$ kJ mol$^{-1}$. An example calculated using the 1st order model of the hydrolysis reaction is shown (below).

This experiment demonstrates the rapid ability of the THT Micro Reaction Calorimeter to determine both the kinetic and thermodynamic properties of a reaction. In traditional calorimeters used for this kind of measurement the time taken for the measurement is very much higher because of the long equilibration time required when changing temperature. The Micro Reaction Calorimeter only requires 30 minutes for full equilibration after a temperature change. The rapid equilibration of the μRC reduces the time taken for this type of experiment from a number of days on previous calorimeters to 3 hours on the Micro Reaction Calorimeter.

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