Syrris Ltd.
27 Jarman Way, Royston, Hertfordshire
SG8 5HW, United Kingdom

T: +44 (0)1763 242555
F: +44 (0)1763 242992
E: info@syrris.com
W: www.syrris.com

Syrris Limited
Registered office as address
Company No. 6976516

Power Compensation Calorimetry study of Acetic Anhydride hydrolysis using Syrris Atlas Calorimeter

Author: Maxime Drobot
Technical Application Leader

Date: 18th July 2013

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1 Introduction

The study was performed by a big flavour and perfume manufacturer.

1.1 System configuration

The Atlas Calorimeter system was used in its standard configuration (Figure 1)

Figure 1

1.2 Experiment details

The studied reaction was the hydrolysis of acetic anhydride in presence of a catalytic amount of sulphuric acid.

To an Atlas Jacketed Vessel containing 650ml water and 2g sulphuric acid, 50ml of acetic anhydride was added during 30min using the Atlas Syringe Pump.

The experiment was fully automated using Atlas 1.4 Software. The results were analysed using Atlas Reporting Software.

2 Hydrolysis of Acetic Anhydride

2.1 Data processing

CSV file “Ac2O_Water-30min-110613” supplied by Firmenich was processed using Atlas Reporting.

The following experiment details were filled in (Figure 2):

- Initial reactor fill volume: 650ml.
- Reactor fluid: Water.
- Added fluid: Acetic anhydride (*Note: this fluid type is not available by default and has been added to the fluid list prior to the data analysis*).
- Temperature of the added acetic anhydride was assumed to be 25°C.

- **Figure 2**

2.2 Results

Atlas Reporting software automatically generated the two following Calorimetry Graphs:

- Overall Power (Figure 3)
- Enthalpy (Figure 4)

Figure 3

Figure 4

The Overall Power graph (Figure 3) gives us the following data:

- **Generated Overall Power** : +17W

This shows that the reaction is exothermic, as expected.

The Enthalpy graph (Figure 4) provides us with the following data:

- **Overall Enthalpy**: 32.11kJ.
  This is the total enthalpy measured during this experiment.
- **Addition**: 1.20kJ.
  This is the enthalpy generated by adding a reagent at room temperature to a reaction mixture at a different temperature.
- **Reaction Enthalpy**: 30.96kJ.
  This is the enthalpy of the reaction itself, calculated by deducing the enthalpy of addition from the overall enthalpy. This is the data of interest for the chemist.

2.3 Calculations

From the Reaction Enthalpy obtained in Atlas Reporting, we can calculate the Heat of Reaction.

Acetic Anhydride properties:

- Density: 1.082 $g.cm^{-3}$
- Molecular Mass: 102.09 $g.mol^{-1}$

Heat of Reaction:

$\Delta_f H°$ = -Reaction Enthalpy / Moles of acetic anhydride

And we know that:

Moles of acetic anhydride = Mass of acetic anhydride / Molecular Mass

= (Density x Volume) / Molecular Mass

Therefore:

$\Delta_f H°$ = -(Reaction Enthalpy x Molecular Mass) / (Density x Volume)

= -(30.96 x 102.09) / (1.082 x 48)

$\Delta_f H°$ = -60.86kJ.mol^{-1}

= -562kJ.kg^{-1}

2.4 Comments

The most recent Heat of Reaction (also called Molar Reaction Enthalpy) for the hydrolysis of acetic anhydride listed by the National Institute of Standards and Technology (NIST) is the following:

$\Delta_f H°$ = -56.6kJ.mol^{-1}

This information can be obtained on their website:
http://webbook.nist.gov/cgi/cbook.cgi?ID=C108247&Mask=8

The difference between the Heat of Reaction obtained from the Atlas Calorimeter and the theoretical Heat of Reaction is 4.36kJ.mol^{-1} (7.5%).