Micro Reaction Calorimetry:

Newer Applications for the Chemical & Pharmaceutical Industry

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All chemical, physical and biological reactions are accompanied by heat change. These reactions, though sometimes subtle, can be measured using calorimetry. This poster aims to show capabilities of the THT µRC [micro Reaction Calorimeter].

The calorimeter consists of a sample and reference cell designed to accomodate 2ml disposible HPCL glass vials. Optional pressure cells rated to 10bar are also available.

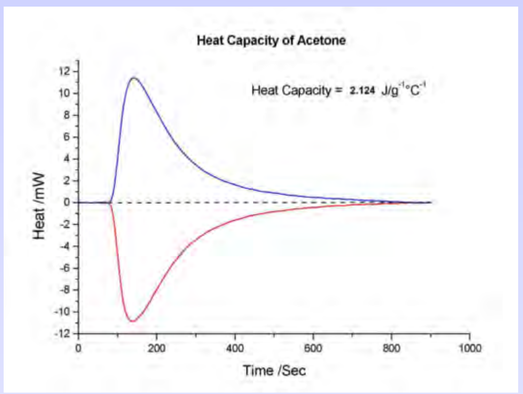
Titration measurements can be made using the automated syringe tower delivering µL injections to the magnetically stirred sample vial.

The in-built pelteir allows for experimental temperatures in the range -5°C to 150°C. Scan rates up to 2°C/min are also feasible.

Heat Capacity Measurement

Measurement of heat cpacity is an integrated function of the µRC. Cp is determined by directly measuring the amount of heat required to shift the sample temperature

A small temperature step (usually in the order of 0.5-1°C) is applied to the system and the heat is measured. The experiment is then repeated in reverse to verify the measurement. The results from each shift direction are averaged to give the final result. Measurement with empty vials was conducted first to ensure that any differences between the heat capacity of the vials is accounted for.



Results

The data in the table below show the results from Cp measurement of pure materials. The values all agree well with the literature data which was obtained from NIST.

	Heat Capacity (Measured)	Heat Capacity (Literature)	Error%
Acetone	2.157	2.130	1.2%
Toluene	1.722	1.704	1.1%
Soya Oil	1.970	1.970	0.0%
$NaCl_{(s)}$	0.864	0.854	1.2%

The largest error was 1.2% which shows the accuracy of the instrument using sample quantity 1g or less.

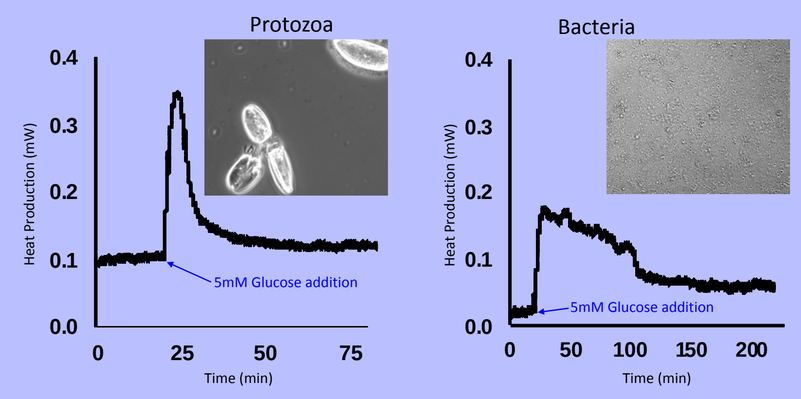
This very simple approach makes the THT µRC ideal for rapid (less than 1 hour) measurement of heat capacity of liquids, solids or mixtures.

Heat Production from Microbes

The sensitivity of the µRC is such that microbial actvity can be accuratley monitored. Rate of heat production of rumen microbes was sucessfuly studied at 39°C in the calorimeter. Rumen fluid was collected from Jersey cows and the cell suspension (1 mL) added to the sample vial against a reference cell filled with water (1 mL).

Heat Production from Microbes

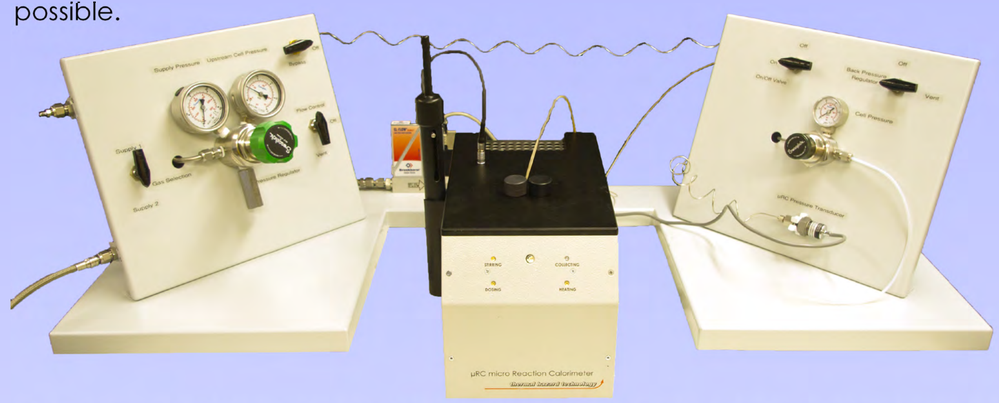
The titration syringe was used to deliver 250 µL of 5 mM glucose.



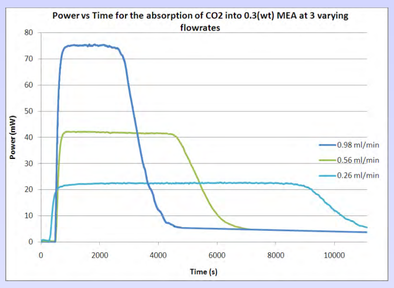
Heat production measured at 1second intervals. Data courtesy of Timothy J. Hackmann, The Ohio State University. USA.

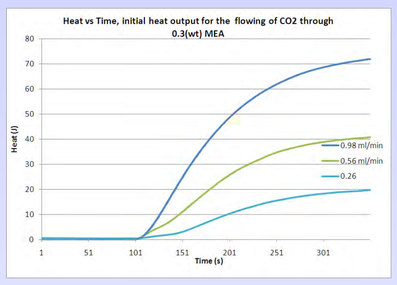
Heat of reaction under controlled gas flow

The Gas Flow Option (GFO) gives added versatility to the instrument making measurement of the heat of reaction at different flow rates and gas pressures possible.



The option has been used to good effect to study Carbon Capture involving the exothermic reaction that occurs when CO_2 gas is absorbed by an amine solution. The GFO consisits of a flow controller to regulate the flow rate of CO_2 into the cell containing the amine solution. Weighing the vial before and after the test is used to calculate the rate of CO_2 update.





Methylethanolamine (MEA) was added to water in 30% concentration by weight CO_2 absorbtion was monitored at three different flow rates.

Enthalpy calculated by;

$$Enthalpy\ of\ Absorption = \frac{Heat}{Number\ of\ moles\ of\ CO_2\ absorbed}$$

$$\Delta H_{abs} = \frac{222.4\ J}{\left(\frac{0.12}{44.01}\ mol\right)} = 81.55\ kJmol^{-1}$$

Sample	Flow rate of CO_2 feed (ml/min)	CO_2 Absorbed (g)	CO_2 Absorbed (mmol)	Energy released (J)	Enthalpy (kJ/mol)
MEA 0.3 (wt)	0.98	0.12	2.7	222.4	81.55
	1.11	0.12	2.7	226.0	82.87
	0.56	0.11	2.5	209.0	83.60
	0.26	0.12	2.7	224.0	82.13
Average	*0.73*	*0.12*	*2.7*	*220.4*	*82.54*

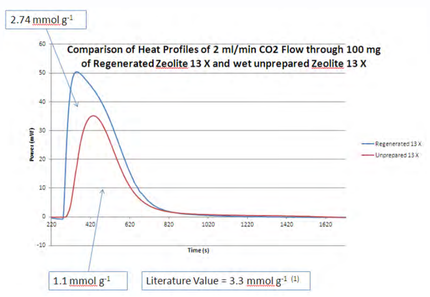
The new Gas Flow option accurately controls CO_2 dosing and allows direct calculation of CO_2 loading in the solvent. The ability of the µRC Micro Reaction Calorimeter to quickly carry out heat of absorption and heat capacity measurements using micro litre volumes of reagents makes it ideal for routine carbon capture studies

Adsorption: CO2 and N2 Gas Flow Through Zeolite

The Micro Reaction Calorimeter from THT can perform isothermal enthalpy measurements on the exothermic reactions that occur when gas is absorbed by Zeolites or other adsorbents.

A comparison of the adsorption capacity of an adsorbent with saturated or unsaturated active sites can be made. Using the Zeolite without prior preparation means that it will already have adsorbed atmospheric species and thus its adsorption capacity will be lower than that of regenerated Zeolite. This is demonstrated in the following graph.

In the two tests shown, 100mg of regenerated Zeolite was measured for heat of adsorption in the µRC with a constant CO2 flow. The test was then repeated with 100mg of unprepared Zeolite. As expected, the unprepared Zeolite with partially-occupied active sites had a measurably lower adsorption capacity than the regenerated Zeolite.



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