Measurement of the volumetric heat capacity by volume of fluids under controlled pressure

Introduction

The measurement of heat capacity is one of the classic measurements carried out by differential scanning calorimetry. While conventional DSC once can measure heat capacity with a relative uncertainty of 5 to 10%, the Ultimate Micro Calorimeter (UMC) developed by Calneos can achieve an uncertainty of close to 1%, or even better under optimum conditions. To achieve this, the Ultimate Micro Calorimeter (UMC) is highly sensitive, regulates temperature very precisely and has large-volume cells to maximise the thermal signals measured.

In addition to the reusable batch vessels, the Ultimate Micro Calorimeter can use volumetric vessels. These are completely filled by injecting the fluid to be analysed. This has 2 main advantages:

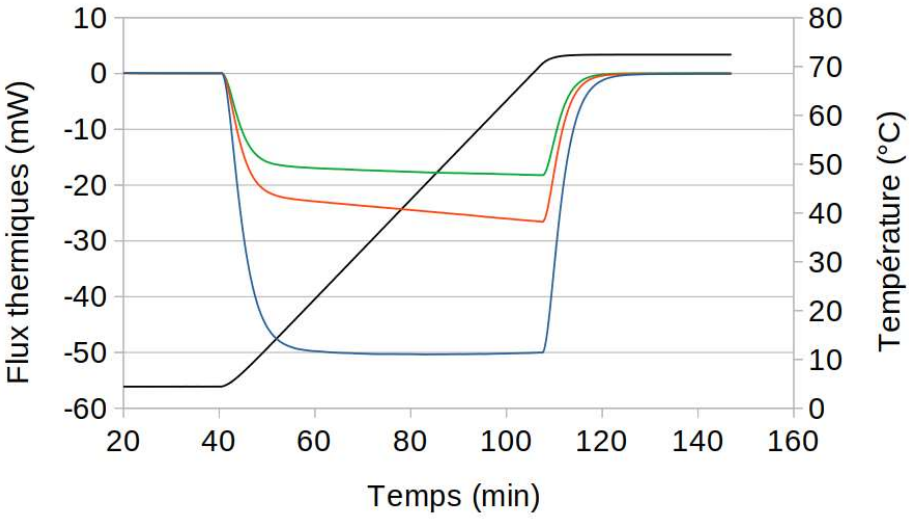
- the first is to avoid any evaporation or pressure variation in the measurement vessel during the temperature ramp
- the second is the possibility to carry out simple measurements under controlled pressure up to one hundred bars.

Experimental protocol

The volume vessel is filled by injecting approximately 3 mL of the liquid sample to be analysed with a syringe. This volume fills the useful volume of the vessel (0.7 mL) and the filling tubes. A slight counter-pressure is applied to the sample at the inlet (around 2 bar) to hold it during measurement and allows the sample to expand during the heating ramp.

The Ultimate Micro Calorimeter was programmed as follows: a 40-minute isotherm at 5°C, followed by a heating ramp between 5°C and 73°C at 1°C/minute, and another 40-minute isotherm at 73°C. A total of 4 experiments of less than 2.5 hours were carried out: with water, absolute ethanol, cyclohexane, and a blank experiment that was subtracted from the other thermograms. The thermograms obtained are shown below.

The heat flow measured is proportional to the volumetric heat capacity of the fluid analysed. Simple mathematical processing of the signal obtained enables the calculation of the volumetric heat capacity. The results obtained at 40°C are shown below:



Results

Cp vol. at 40°C (J/mL/°C)	Theoretical	Measured
Water	4,15	4,13
Ethanol absolute	1,99	1,99
Cyclohexane	1,45	1,45

Conclusion

With its dedicated vessel, the Ultimate Micro Calorimeter is a highly effective tool for measuring volumetric heat capacity. It enables measurements under controlled pressure over a wide temperature range (-30°C to 160°C) with a great accuracy.

The Ultimate Micro Calorimeter also has several types of vessel: batch, volume, mixture, reaction, etc.

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