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AN AUTOMATIC TWIN CALORIMETER FOR FLUID SYSTEMS

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A computer-aided calorimeter for the region of -15° to 180°C and with a resolution $2 \cdot 10^{-5}$ K is described. The reactive volume may be varied from 10 to 100 ml.

Keywords: automatic twin calorimeter

Introduction

This calorimeter is based on a new polyfunctional concept and can be used to investigate heat effects produced by various chemical reactions and physical processes [1]. This instrument eliminates disturbing factors on the basis of a double-chamber design. The desired constant temperature is guaranteed by a special precision thermostat. The liquid media used in the bath are water for measurements in the normal region of 10° to 85°C, a mixture with glycol for the adjacent region down to −15°C, or silicon oil for higher temperatures to 180°C.

Experimental

The equpment is shown schematically in Fig. 1. The container for the liquid thermostated bath was designed in such a way as to prevent regions of insufficiently circulated liquid, i.e. all parts of the bath being subjected to forced circulation. This is achieved by the design of the container as well as by the pump (4) which is driven by magnetic coupling from underneath the bath. In order to minimize the influence of ambient temperature on the temperature of the thermo-

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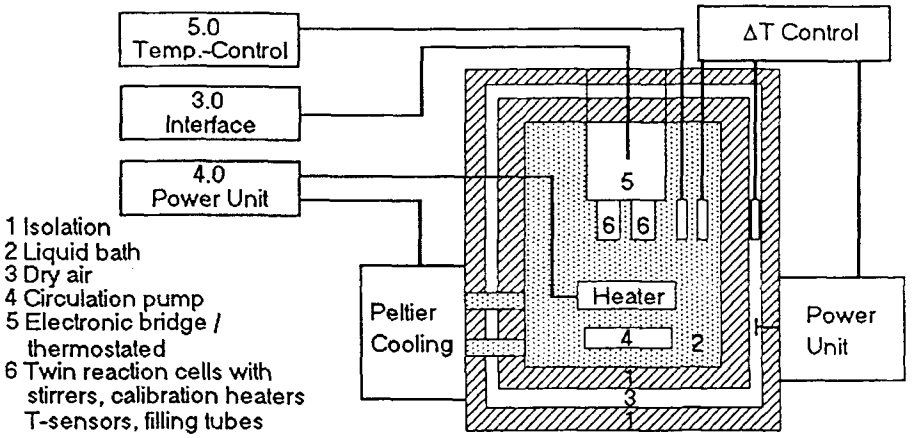

Fig. 1 Schematic diagramm of twin calorimeter

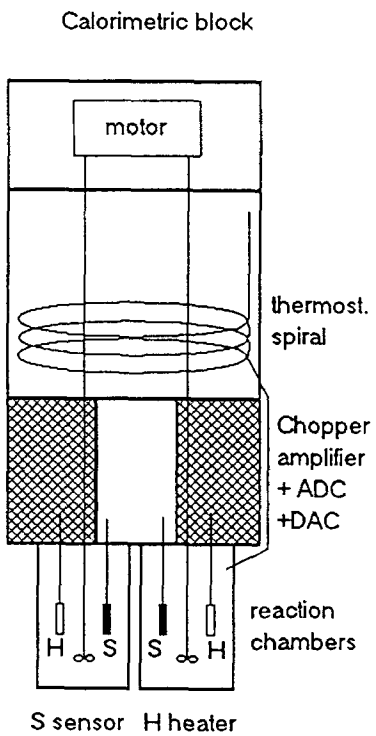

Fig. 2 Schematic diagram of reaction chambers and heating block

stat, a double-wall insulation was incorporated. The intermediate space kept at a suitable temperature by a controller.

If it is necessary to cool the bath, it is connected to a controlled Peltier block, the warm side of which transfers the heat to the environment. If required, the power unit of the Peltier block (4.0) supplies the heater of the bath with energy. The temperature of the thermostat is constant within ±0.15 mK for several hours, and less than 1 mK per day.

All functions of the calorimeter are oprerated by a personal computer which feeds its data by a bus driver into an interface 3.0). In the inverse direction the measured data from the calorimeter are returned to the computer. The interface is also responsible for the temperature-control unit (5.0) of the bath. As temperature sensors, several silicon sensors SAS 1000 and YSI 46007 are used. The heater of the bath liquid was designed in such a way as to achieve efficient and fast heat exchange.

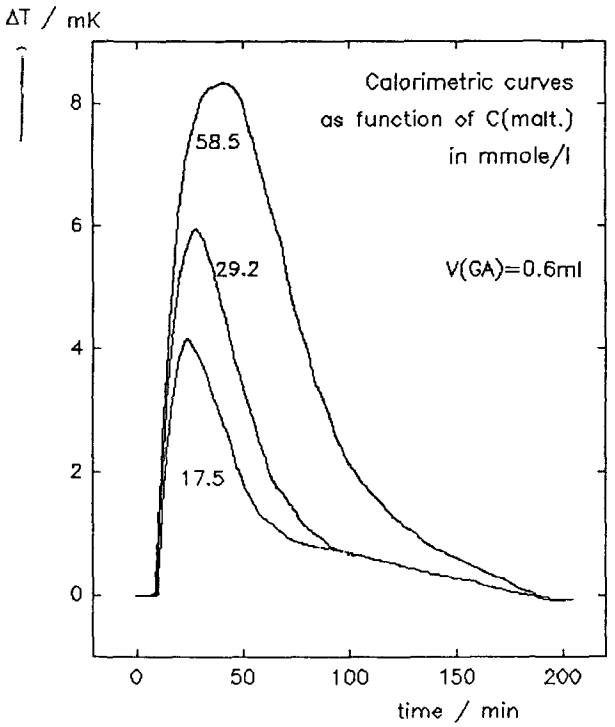

Fig. 3 a Calorimetric curves for hydrolysis of maltose

Calorimeter measurements are performed in both reaction chambers of the twin arrangement. These are easily connected to the calorimeter block (Fig. 2) which is immersed in the bath. Volumes of the reaction cells can be varied up to 100 or up to 40 ml. Each cell incorporates a calibration heater, a stirrer and a tube for the second reaction component, as well as a tube for degassing or an inert gas. The second reactant is thermostated in a spiral and can flow into the cells according to a selected programme. Stirrers have synchronous revolution with digitally controlled rate of revolution.

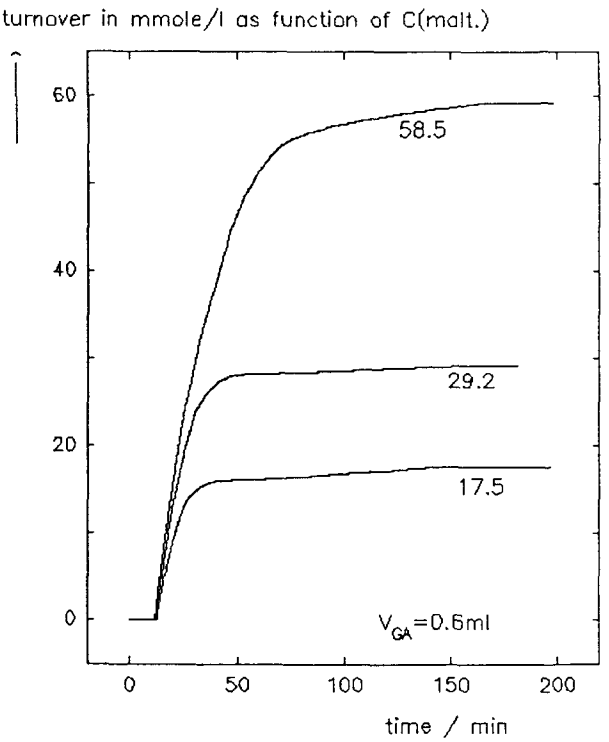

Fig. 3.b Calorimetric curves for hydrolysis of maltose

Each temperature sensor is one branch of a multiple-bridge circuitry. Compensation of the bridge is performed by the computer via an 18-bit DA-converter. The difference voltage is pre-amplified with the aid of a chopper-stabilized amplifier located directly above the reaction cells. Due to this arrangement the bridge electronics work under strictly constant temperature. An 18-bit AD-converter feeds the signal into the computer. Applying the multiple bridge curcuitry it is

possible to measure separately the temperature of the bath, the temperature in each reaction cell, and the difference between both cells.

The calorimeter reaches a temperature resolution of about $2 \cdot 10^{-5}$ K. The signal disturbance is lower than $5 \cdot 10^{-5}$ K.

All parts of the calorimeter, with the exception of the computer, are contained in a compact casing. Loading and emptying of the reactors are easily performed. A titration device can be installed. The software for the computer contains the following variants of data evaluation as a function of time: heat sum; heat production; reaction or process rate; reaction rate constants; stoichiometric conversion.

Figures 3a and 3b illustrate some results obtained with this calorimeter. The enzymatic hydrolysis of maltose by glucoamylasis is a slow reaction. The typical effect of concentration of this bio-catalyst is clearly visible.

References

1 H. Anderson, H. Lubenow, W. Klingner and K. Heldt, Thermochim. Acta, 187 (1991) 211.

Zusammenfassung — Es wird ein computergestütztes Kalorimeter für den Bereich –15° bis 180°C und einer Auflösung von 2×10^{-5} K beschrieben. Das Reaktionsvolumen kann einen Wert zwischen 10 und 100 ml betragen.