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A NEW MICRO REACTION CALORIMETER

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Abstract

Traditional reaction calorimeters require highly trained operators and the use of a large sample size (10 ml to 1000 ml) which is incompatible with highly reactive substances. A new calorimeter with a 2 ml or less sample size lends itself to the determination of reactivity of materials for process safety and to the determination of safety of materials shipped in compartment tank cars. The TC-100 Titration Calorimeter meets these requirements. A cost effective instrumentation of a differential cell design provides high sensitivity heat flow measurements. Automatic liquid injection and continuous mixing provides information on the heat of reaction and its rate. A portable instrument with small sample size allows for rapid temperature equilibration and safe handling of very reactive materials.

Keywords: hazards, microcalorimeter, physiology, portable, safety, titration

Introduction

The need for a more portable 'in the field' calorimeter has become more obvious in recent years. This need is a results of applications for which calorimetry has been shown to have a distinct value. One of these applications, the hazardous and safety concerns for compartmentalized tank car shipping, has been reported and discussed at this conference by T. C. Hofelich [1, 2] and others. A second application, 'Plant Physiology and Ecology,' has been reported at this conference by L. D. Hansen [3, 4] and others. Many other applications apply as well.

Several features assist the utility of a portable calorimeter. A weight of less than 100 kg and a size that can easily sit on a lab bench are important. A disposable 2 ml sample container like the crimp top autosampler vial make the users job economical and simple. These vials may contain solid or liquids and may or may not require mixing. A standard Hamilton 100 µl syringe can inject a second reactant if necessary.

Experimental

Instrument design

The Model TC-100 (Fig. 1) calorimeter is a twin cell heat conduction style. The sample containers are standard 2 ml crimp top or screw top glass vials available

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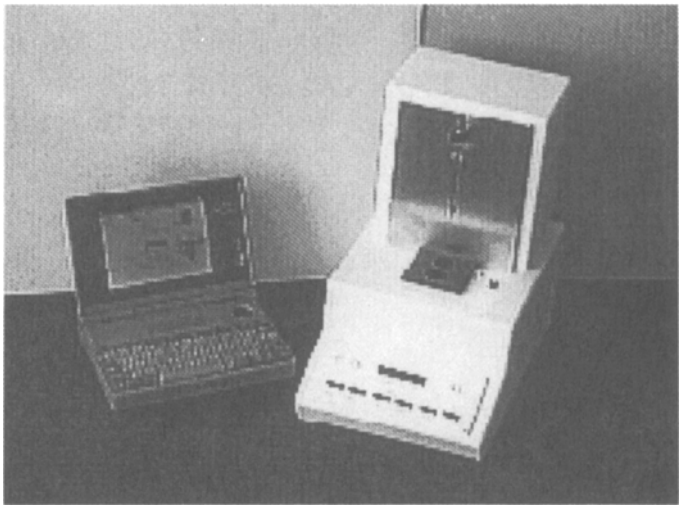

Fig. 1 TC-100 Micro Reaction Calorimeter with a laptop

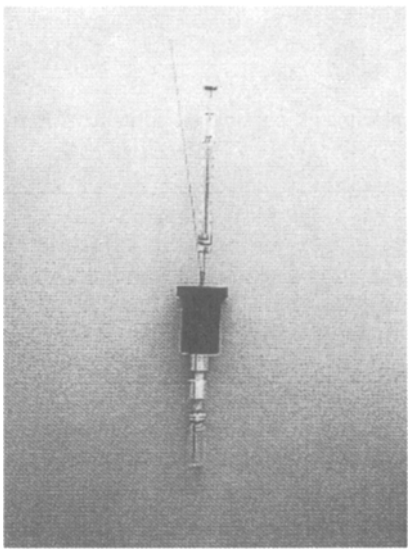

Fig. 2 Sample assembly with syringe before insertion

from several manufactures. The syringe and vial assembly (Figs 2–3) are lowered into the front of the unit and allowed to equilibrate before starting the measurement.

Calibrations are done routinely with joule heating (Fig. 4) in the cell wall assembly and checked with acid base neutralization (Fig. 5). Since the TC-100's data output is RS232C, a laptop or other Windows™ based computer can collect data in one window while another application like data analysis can run in another window.

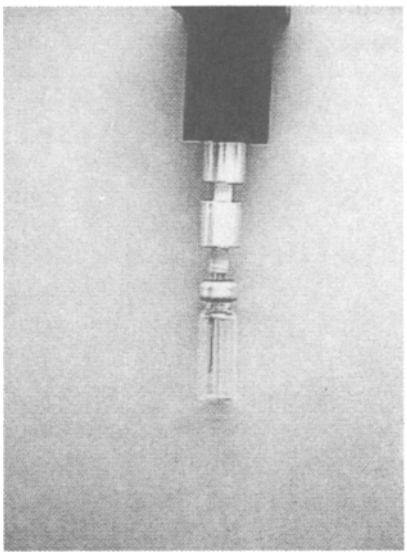

Fig. 3 2 ml vial before insertion

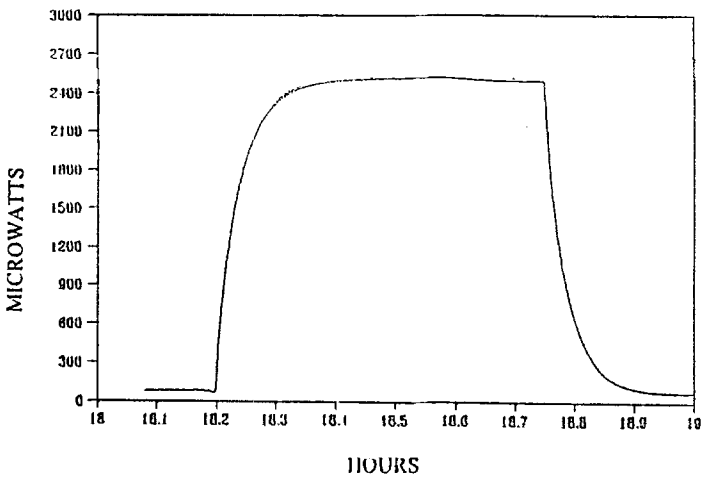

Fig. 4 Electrical calibration

The data format is ASCII and is compatible Microsoft's Excel™ and Microcal's Origin™.

Calorimetric measurements

The baseline stability (Fig. 6) is a microwatt over a several hour period. When the laboratory room changes 10°C the base line (Fig. 7) moves several microwatts.

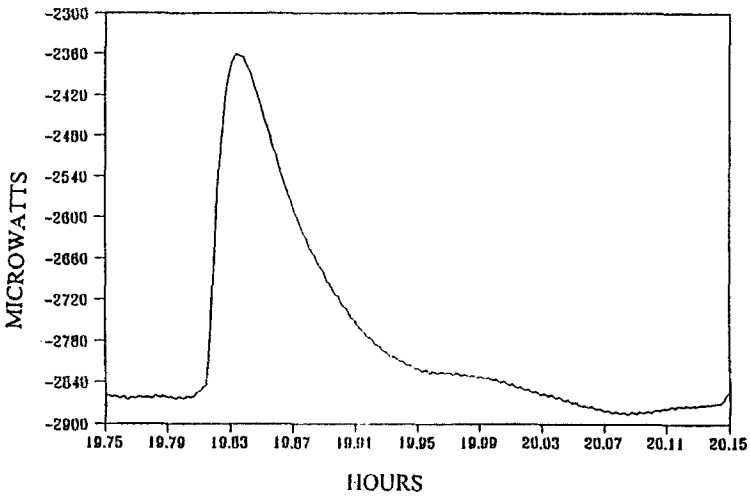

Fig. 5 Chemical calibration

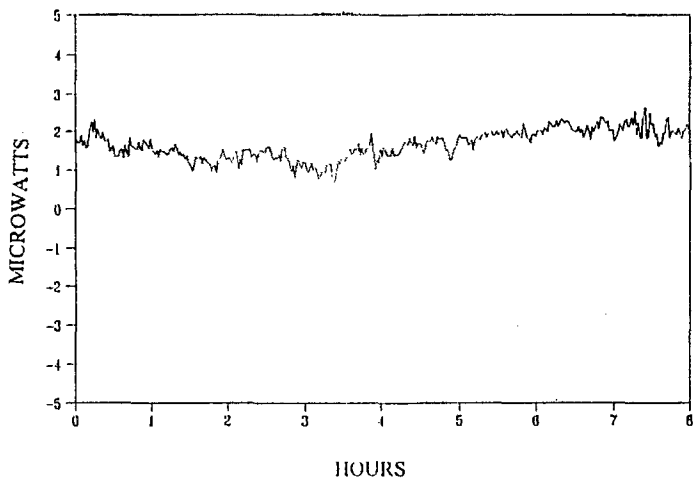

Fig. 6 Baseline stability

A typical acid into base injection has the standard heat conduction shape (Fig. 5). The area under the curve represents the heat of this reaction.

Results and discussion

It has been shown [5] that multiple pulses or injections can be deconvoluted to allow faster injection repetitions than the time constant of the instrument would seem to permit. A variety of experiments can be run with this unit with traceability to a chemical standard like TRIS titrated with an acid. A solid can be loaded into

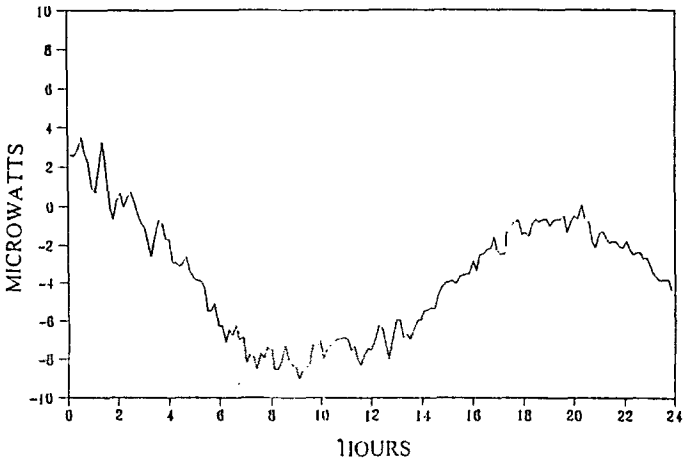

Fig. 7 Baseline stability as the room varies 10°C

the bottom of the vial and dissolved with a syringe injection. A single injection can be made or a stepwise titration can be made to find both equilibrium constants and enthalpy. Plant or other living materials can metabolize or chemicals can decompose in the vials for a measurement.

Summary

To meet the need for a more portable 'in the field' calorimeter a new instrument has been developed. The micro reaction calorimeter has a distinct value for chemical hazard, safety testing, and for plant physiology research. A small foot print for the instrument has been achieved. Disposable glass 2 ml vials accent the economical value of the unit. The small sample can be injected with another liquid and mixed for best results. Various temperatures and conditions can be set for sample testing from the instruments front panel or with a laptop computer while it is collecting data.

References

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