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A DIFFERENTIAL SCANNING CALORIMETER FOR EDUCATIONAL PURPOSES

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SUMMARY

A new Differential Scanning Calorimeter is introduced, which has been developed specially for educational purposes. A discussion is given of the various boundary conditions connected to the development of this instrument and the resulting construction is described. The specifications of the instrument are presented.

INTRODUCTION

In the past decade, the use of thermal analysis for R&D and QC purposes has grown very rapidly. Based on the potential of the various thermal analysis techniques, DSC, TGA, TMA, DMA etc., it is expected that within some years these techniques will be used within almost every laboratory. Therefore, a proper education in thermal analysis nowadays is a necessity for every research chemist and laboratory assistant.

Unfortunately however, thermal analysis is hardly ever placed on the curriculum. Clearly, education considerably lags behind the developments within industry.

One of the reasons for this clearly is the lack of proper TA instrumentation for educational purposes. Commercially available thermal analyzers mostly do not fit into the low budgets of educational institutes, moreover, they are not optimised also for educational purposes.

To stimulate a rapid introduction of thermal analysis within education (one of the aims also of the ICTA Education Committee) we decided to develop a range of "educational thermal analyzers". As DSC is by far the most frequently used technique within thermal analysis the first instrument in this line is an Educational DSC.

BOUNDARY CONDITIONS

Educational purposes require very specific development boundary conditions. For this instrument these conditions were defined as follows:

Specifications

Though for educational purposes specifications may lie on a somewhat lower level than required for R&D and QC applications, the instrument clearly should be suited for more than only demonstrating thermal effects in samples. First, the temperature range should be rather broad, covering nearly all normal applications, also those sub-ambient. Secondly, the instrument should allow quantitative measurements to be performed, like e.g. purity and kinetic determinations. Finally, the sensitivity of the instrument should be rather high, allowing measurements to be performed with only milligrams of sample.

Price

In order to reach the defined goal, a rapid introduction of thermal analysis techniques within education, the price of the instrument should fit into the very limited budgets of educational institutes. This boundary condition requires a special modular design of the instrument guaranteeing a fast and reliable production and a low service proneness. Especially when specifications must remain on a professional level, requiring the use

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of expensive high quality components, reductions of the cost-price are to be found in assembly-, test-, service- and sales costs. Market research in the Netherlands revealed that an end-user price in the range of US$ 5000 - 6000 would still fit into the educational institutes budgets. It is assumed that in other countries this situation is similar.

Quality

The quality of the instrument should not only be reflected in its thermal specifications, but also in what is called "user friendliness". Especially in education, where the instrument is used by a great number of very different persons, not all having special skills in working with instruments, these "attacks" should be survived by it. This requires a special rugged sensor design and built-in safety circuits. At the other side, also care must be taken to protect the user. A further requirement in this field is the ease of operation. As most students only work with the instrument during not more than half a day, it should not take more than 30 minutes to familiarize with it.

Extra features

The following extra features should be mentioned:

flexibility

Operational errors should easily be correctable. An example is the possibility to change measurement parameters while the instrument is running, without disturbing the measurement.

dimensions

The instrument should comprise only one casing, not exceeding a size of 40x40x20 cm.

computer control

In education, computers already are very popular and many students have a special affection to work with them. Besides manual control, the instrument therefore should have full computer control as a standard and access to the measured data should be easy.

DESCRIPTION

The resulting instrument is built up of four basic units:

1. measuring cell
2. instrument control/data acquisition board
3. power supply board
4. signal amplifier

ad. 1. The measuring cell contains a low-weight aluminium furnace, resistance wire heated. The furnace is surrounded by a heat exchanger which enables high cooling rates. Temperature measurement and control is by a Pt 100 resistance thermometer. The very rugged heat flow sensor is of the disc type, with the thermocouples attached from below.

ad. 2. All functions of the instrument are controlled by a Siemens 80535 microcontroller. The use of a microcontroller in stead of a microprocessor has the great advantage of considerably reducing the number of components, in this way enhancing the simplicity of the electronic circuitry. Both A/D and D/A conversion proceed with a resolution of 16 bits.

ad. 3. The furnace is heated by means of DC power. Special care has been taken to eliminate as much as possible the influence of mains perturbations, as this voltage is, particularly in educational institutes, far from perfect. On this board, also the various safety circuits are located.

ad. 4. The measured voltage is amplified by means of a newly developed signal amplifier, mounted very close to the measuring cell, to avoid noise pick-up. The signal amplifier features an electrical noise level below 50 nV peak-peak with a band width of 1 Hz.

SPECIFICATIONS

The thermal specifications of the instrument are listed below:

temperature range	-50 °C - +400 °C
heating rates	1 - 20 °C/min
thermal noise	approx. 25 uW
signal time constant	approx. 4 sec.
base line drift 20-400 °C	< 5 mW

A typical measured curve is presented in fig.1.

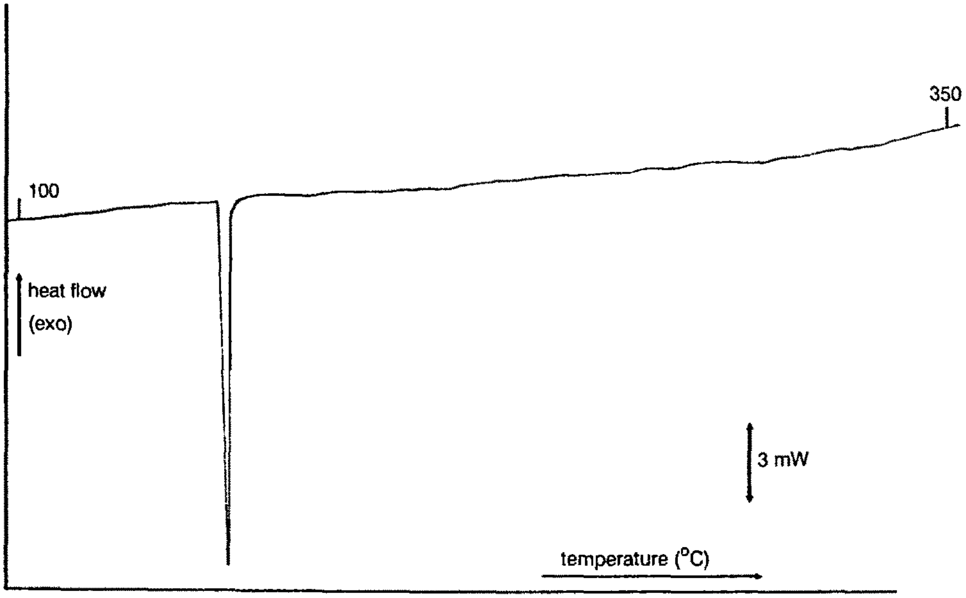

Fig. 1. DSC curve of Indium.

Scan parameters:

heating rate	10 °C/min
temperature range	100 - 350 °C
range	30 mW full scale
sample weight	4.73 mg
atmosphere	static air

OPERATION

The instrument can be operated both manually and under computer control.
In the manual mode, a small numerical keyboard serves for input of scan parameters. The actual cell temperature is presented on a large LCD display (temperature read-out is possible both in °C and in K). A second LCD display is used for read-out of scan parameters and for display of instrument status and error messages. The instrument has an analog output, enabling the measured signal to be registered on a normal chart recorder. A temperature marker signal can be superimposed onto the measured signal, in this way enhancing the ease and precision of temperature determinations.
In the remote (computer) mode, the instrument is controlled by an IBM compatible computer, using the MS-DOS operating system. Operation proceeds using a serial (RS 232) interface. For this purpose a software package based on MS-Windows has been written. With this very user friendly software package, instrument control proceeds by defining a measurement menu, consisting of up to three different temperature-time profiles and which can be stored on disk. After having defined the sample parameters, the measurement is started and the resulting curve is displayed real-time onto the screen. The curve can be stored on disk and at a later time basic calculations, like temperatures and peak areas can be performed. Access to the measured data is very easy, facilitating their input into other software packages like Lotus 1,2,3 etc. Using the parallel interface, the curves can be output on a graphics printer.

EXPERIMENTAL THERMAL ANALYSIS

To enhance a rapid introduction of the student into the potential of Differential Scanning Calorimetry a textbook has been written entitled "Experimental Thermal Analysis".
In this book, first the backgrounds of the various thermal analysis techniques are treated and examples are given of the applications within various branches of chemistry, pharmaceutics and physics. In a next chapter, the instrument and its operation are treated. A main part of the book is devoted to a number of selected, very illustrative experiments. Of each experiment, the underlying theory is given, the experiment is thoroughly described and the resulting curve is explained. In this way the student will be able to familiarize with thermal analysis and its advantages in a relative short time and with a minimum of teacher assistance.