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THE DSC12E, A LOW-COST DIFFERENTIAL SCANNING CALORIMETER

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The DSC12E is a low-cost Differential Scanning Calorimeter, specially developed for quality control- and 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 discussed with the aid of some illustrative experiments.

Introduction

A low-cost differential scanning calorimeter has been developed, specially suited for quality control and educational purposes [1]. The development boundary conditions of the instrument were defined as follows:

Specifications

Though, for quality control and educational purposes specifications may lie on a somewhat lower level than required for R&D 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.

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Price

In order to reach the educational- and small-industrial market, the price of the instrument should be as low as possible. This boundary condition required 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 of expensive high quality components, reductions of the cost-price are to be found in assembly-, test-, service- and sales costs.

Quality

The quality of the instrument should not only be reflected in its thermal specifications but also in what is called 'user friendliness'. Especially for educational and routine industrial purposes, where the instrument has to be 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, care must be taken also to protect the user.

A further requirement in this field is the ease of operation, it should not take more than 15 minutes to familiarize with the instrument.

Other keywords during the development of the instrument were flexibility and computer control.

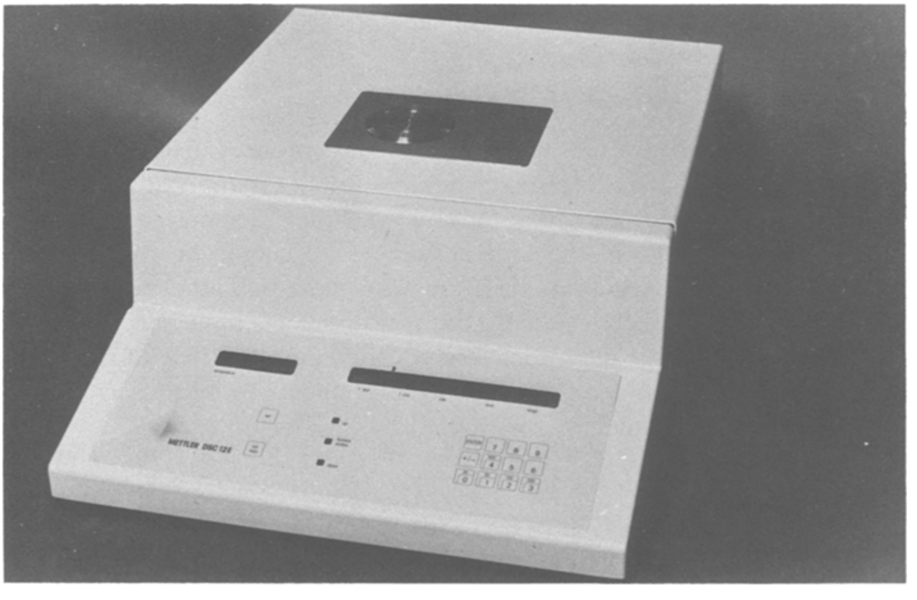

Fig. 1 DSC12E

Description

An overview of the DSC12E calorimeter is given in Fig. 1.

The total instrument comprises one casing only, with dimensions 50×40×17 cm (l×w×h).
Clearly the frontpanel with the simple keyboard, the large LC displays and the lid of the measuring cell can be distinguished.

According to the specified boundary conditions the intrument is of a full modular construction.
All control functions are integrated on only one printed circuit board. This board contains the microcontroller, memory, A/D conversion, the displays and the keyboard.

The measuring cell assembly consists of the furnace-sensor assembly and the cooling jacket. The furnace is constructed of aluminium and resistance wire heated. The sensor is of the disc type and the thermocouples are spot-welded at the lower side. The furnace is surrounded by the cooling jacket, which is liquid cooled. Normally water is used, but as easily e.g. cooled ethanol can be applied as cooling agent, facilitating sub-ambient operation.
The signal amplifier is located as close as possible to the measuring cell. In this way noise pick-up is reduced. The amplifier has an electrical noise <40 nV peak-peak and a small time constant.

Operation

The instrument can be used in two modes, the manual mode and the remote mode.
In the manual mode, the measurement parameters are input by the numerical keyboard at the front panel. A measurement may comprise both a dynamic part and an isothermal part. All measurement parameters are continuously shown on one of the displays.
The output of the measured curve can proceed in two ways, on an analog chart recorder, or on a matrix printer.
When the curve is output on a chart recorder, a temperature marking signal can be superimposed onto the measurement signal, allowing easy determination of temperatures.

When the output is on a matrix printer, first a header is printed, in which information on the sample can be specified and in which also the measurement parameters are given. The curve then is printed on the remaining part of a normal sheet of paper.

When working under computer control, operation proceeds by a software package based on MS-Windows. This software package has been developed employing the same philosophy as the instrument itself: it features a great user-friendliness and flexibility.
The package, with the name TA89E, allows complete control of the instrument. Also complex temperature-time profiles can be defined, up to a maximum of eight.
The measured curves can be displayed on-line onto the screen, while simultaneously, operations on previously measured curves are performed. The curves can be stored for later evaluation.
Up to eight curves can be displayed simultaneously onto the screen allowing easy comparison of measured curves. The curves, together with the results of he operations and calculations performed can be output on a matrix printer.

Performance

The thermal specifications of the instrument are summarized below:

temperature range	−50°C up to +400°C
temperature precision	0.25°C
temperature reproducibility	0.1°C
heating rates	1–20 deg/min
thermal noise	approx. 20 μW
signal time constant	approx. 3 sec.
base line drift 20–400°C	<5 mW
base line reproducibility	<1 mW
gas atmosphere	static air
sample pans	aluminium, 40 μl volume
dimensions	50×40×17 cm (l×w×h)
weight	15 kg
power consumption	300 W max.

The performance of the instrument can be illustrated best with some examples.

In Fig. 2, the DSC curve is given of 11.1 mg of PET.
This well known curve clearly shows the baseline behaviour of the instrument and its ability to work with milligrams of samples. The various features of the TA89E software package are also well demonstrated.

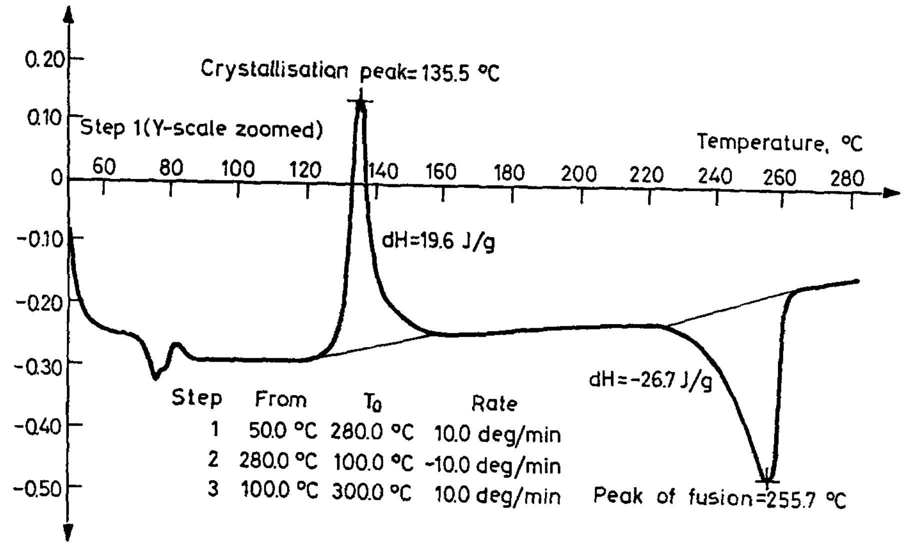

Fig. 2 DSC curve of PET, measured with the DSC12E

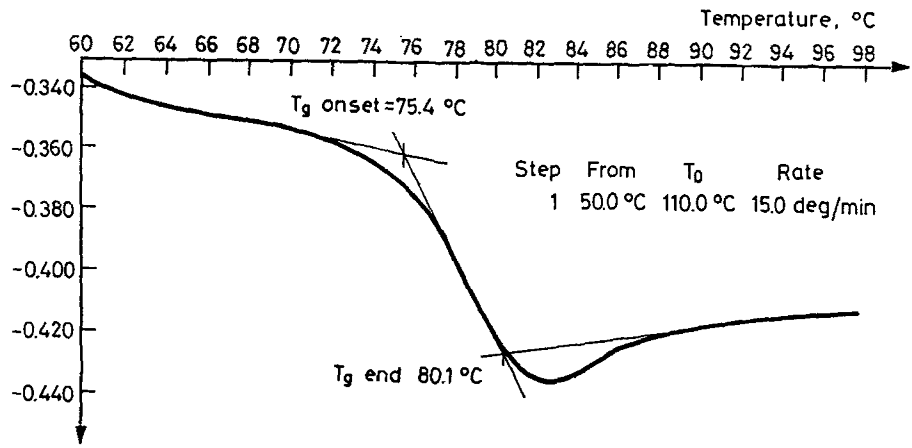

Fig. 3 Glass transition of PET

In Fig. 3, the glass transition of PET is presented, measured with 10.6 mg of sample and a heating rate of 15 deg/min. This curve clearly indicates the low noise of the DSC12E.

The DSC12E calorimeter will be commercially available by Mettler-Toledo, Greifensee, Switzerland.

The practice of Thermal Analysis

To stimulate the introduction of Thermal Analysis into education a textbook has been written, entitled 'The practice of Thermal Analysis'. This book gives the backgrounds of the various techniques and examples of applications within various branches of chemistry and physics. In a comprehensive way, a number of very illustrative experiments is described which can be performed in a relatively short time.

References

1 G. van der Plaats *et al.*, Thermochim. Acta, 151 (1989) 319.

Zusammenfassung — Das DSC12E ist ein Differential Scanning Calorimeter der niedrigsten Preisklasse, das sowohl für die Qualitätskontrolle im Routinelabor als auch für Ausbildungszwecke entwickelt wurde. Es werden die Spezifikationen und die Rahmenbedingungen dieses Instrumentes erläutert und erste Messungen dargestellt.