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Reconstruction of a Perkin Elmer DSC-2 to a computer-driven calorimeter

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Abstract

A possibility to reconstruct a Perkin Elmer DSC-2 to a computer-driven calorimeter and to use it with self-made software is pointed out. The necessary components and hardware changes are described and a brief outline of the functionality is given.

1 Introduction

The commercially available scanning calorimeters of the new generation are usually equiped with computer based data acquisition systems and user-friendly software for computation of the acquired calorimetric curves.

In many laboratories we can find yet older calorimeters which work satisfactorily but are not state of the art any more, because they have to be operated manually and the elaboration of the results has also to be done by hand. In many of those cases it would be profitable to take the reconstruction of such a calorimeter into account.

We decided to equip our DSC-2 with a user-friendly computer-steering instead of the Tektronix control-unit (2000 program steps, 1000 data points) which steered the calorimeter before.

In the following paragraphs the functioning of the DSC-2 will be described and afterwards the necessary steps for the reconstruction of the calorimeter will be shown.

2 Function principle

The Perkin Elmer DSC-2 is a power-compensated isoperibolic working differential scanning calorimeter. It can be divided into two parts: the digital and the analog part.

The digital part contains the whole electronics necessary to convert the input parameters (T_{Begin}, T_{End}, Heating-Rate) into a voltage (program-voltage) which is proportional to the target-temperature (program-temperature). The regulation circuits of the analog part of the DSC-2 need this voltage as an input quantity while the output quantity is represented by the signal-voltage which itself is proportional to the heat flux into the sample.

To help the reader comprehend the functioning of the digital section, a simplified form of it shall be discussed (see Fig. 1).

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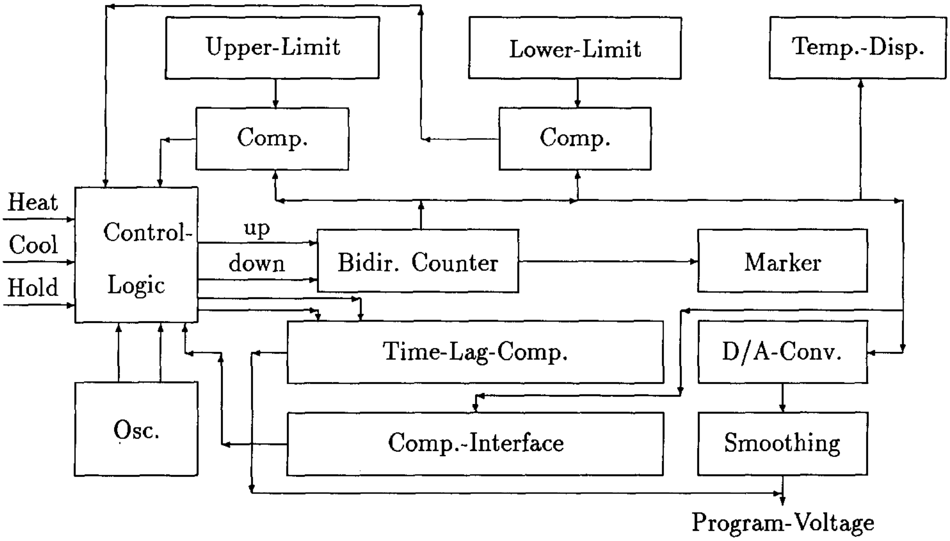

Figure 1: Schematic diagram of the digital section of the DSC-2

The central unit of the digital section contains an D/A-converter with resolution of 15 bits whereby the lowest bit represents a temperature change of 1/30 K. Four bidirectional 4 bit BCD-counters and two additional binary coded lines, connected to the counters via an additional digital circuitry, are supplying the D/A-converter with the digital input information. Therefore the clock signals for the counters steer the output voltage of the D/A-converter just as the program temperature. In addition there are built-in comparators which compare the actual counting with two adjustable limits and cut the inputs of the counters if correspondence is present.

For that, the whole circuitry represents a frequency-steered Counting-Voltage-Converter (CVC) with two inputs (up- and down-counting) and built-in switch off.

The clock signal for the counters, which is created by an oscillator, is divided by dividers downto the needed frequencies. These frequencies can be switched to the inputs of the CVC by step-switches and intend by this an increase respectively decrease of the program-voltage (and therefore of the program temperature too) which is proportional to the magnitude of the input frequency. For this reason the heating- resp. cooling-rates of the DSC-2 are represented by these frequencies, dependent on the free input at the CVC. The inputs of the CVC are opened by logic AND-gates which are connected to the input lines and whose second inputs can be set resp. reset by press-keys. The apparatus-function HEAT is triggered by the opening of the count-up-input as well as the apparatus-function COOL is triggered by the opening of the count-down-input. Both inputs are closed when the isothermal mode is established.

The press-keys for HEAT, COOL and HOLD (isothermal mode) can be distant-controlled by relay, just as the program temperature can be read out by 18 digital lines.

The voltage created by the CVC has to be smoothed out because of its step-form before it can be connected to the regulation circuitry. The smoothing circuit used for this causes

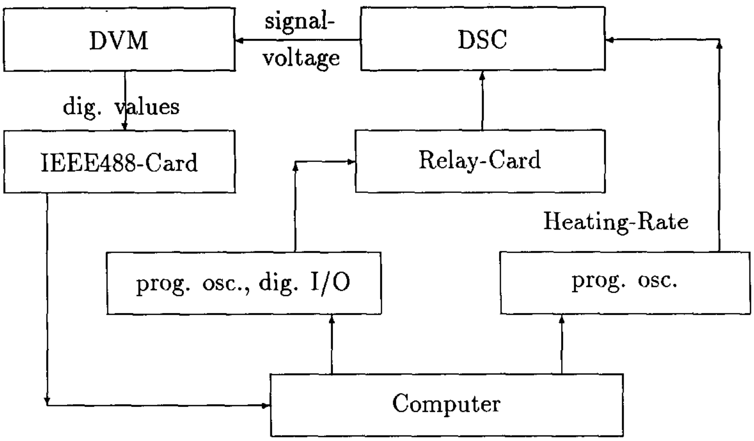

Figure 2: Schematic circuitry of the additional hardware

a time lag in the rise of the program-voltage which depends on the heating- resp. cooling-rate. This time lag can be compensated by adding an additional voltage to the program-voltage, which is proportional to the heating-/cooling-rate (time lag compensation).

3 Hardware changes

To realize a complete computer-steering, the following functions have to be done by the computer:

- steering of the following DSC2-funktions:
  - HEAT, COOL and HOLD
  - setting of the heating- resp. cooling-rate
- reading of the program-temperature
- reading of the heat-flux signal-voltage

The recording of the signal-voltage is done by a $5\frac{1}{2}$-digit DVM which can be manipulated and read out by the computer via IEEE488-Bus. A parallel I/O-Computer card is used to get access to the BCD-coded program-temperature (with 1/30 K resolution) and the functions HEAT,COOL and HOLD were made computer-accessible via a self-made relay-circuit. Finally a programmable oscillator is used to create the steering-frequency for the heating-/cooling-rate and at the same time the necessary time lag compensation voltage is connected via a 12 bit D/A-converter. The basic interconnections of this hardware are shown in Fig. 2.

4 Develloped software

The software necessary to steer the described hardware is able to do complete measuring-cycles by itself. In addition, an evaluation software was written which allows the following standard-computations:

- peak calculations (onset, max/min, area)
- glass-transition calculations (T_g, Δc_P)
- calculation of the specific heat and the enthalpy function

Besides, additional software has been develloped such as

- calculation of the temperature-profile in samples of big size
- desmearing of the apparatus- and sample-influence on the measured signal (see [1])
- calculation of the lamella-thickness-distribution in polymers (see [2]).

References

[1] **J. Schawe, C. Schick** *Thermochimica Acta, this issue*

[2] **M. Alsleben, C. Schick** *Thermochimica Acta, this issue*