Thermochimica Acta 382 (2002) 77–87

thermochimica acta

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Individual solutions for control and data acquisition with the PC

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Received 8 May 2001; received in revised form 14 May 2001; accepted 15 June 2001

Abstract

Individual and flexible solutions for control and data acquisition are an important tool for measurements in scientific research, since very often self-constructed or adapted commercial measuring devices are applied. Software which is purchased together with the instruments is in many cases not suitable for research applications, which require comprehensible procedures with high precision and reproducibility. Suitable programmed commercial solutions are either not available or too expensive. This paper presents the adaptation and automation of a high temperature Calvet-type microcalorimeter. It allowed us to perform programmable drop calorimetry with automated additions of samples under controlled atmosphere and temperature as well as signal acquisition at low cost, which does not require any special knowledge of electronics or programming. We were able to integrate several instruments for measuring and controlling via their communication interfaces (IEEE, I/O-port) by means of the program Laboratory Virtual Instrument Engineering Workbench (LabView, National Instruments). The program runs on a conventional Pentium-PC under Windows98. It allowed us to construct user interfaces for the input of control parameter and observation of running measurements as well as the import of acquired data to the evaluation software HiQ (National Instruments).

Keywords: Calorimeter; Automation; Data acquisition; Sample introducer

1. Introduction

In scientific research, in particular, we often use very sophisticated devices for measurement and control of different parameters, employing self-constructed or greatly adapted commercial apparatus. Nevertheless, we demand modern measurement performances with a high level of automation as well as control and data acquisition via computer. Similar situations occur, if we want to modernise existing measurement devices.

In these cases, we need individual solutions which are, however, either not available in the market or too expensive. For individual and flexible low cost solutions it is necessary to find easily programmable software which runs on conventional PCs. On one hand, it should be able to communicate with serial ports, IEEE-cards, digital I/O-cards, data acquisition cards, etc. On the other hand, it should be easy to import acquired data to individual evaluation software. Beyond that, we need a suitable user interface to enter control parameters and observe running measurements.

This paper describes the adaptation and automation of a high temperature Calvet-type microcalorimeter HT-1000 (Setaram), which is an important improvement to the previous work reported in [1–3]. Our report can give the necessary information together with the hints and ideas to people in similar situations.

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0040-6031/02/$ – see front matter
PII: S0040-6031(01)00739-0

2. Apparatus

A schematic diagram which describes the arrangement of the whole equipment is shown in Fig. 1. In Table 1, the employed instruments and software products are listed which are commercially available are listed. Our report not only focuses on the aspects of the automation of the sample introduction, control and data acquisition but also gives a brief description of the entire equipment.

2.1. Calorimeter furnace

The Calvet-type microcalorimeter furnace is a twin-calorimeter with two separate calorimeter cells,

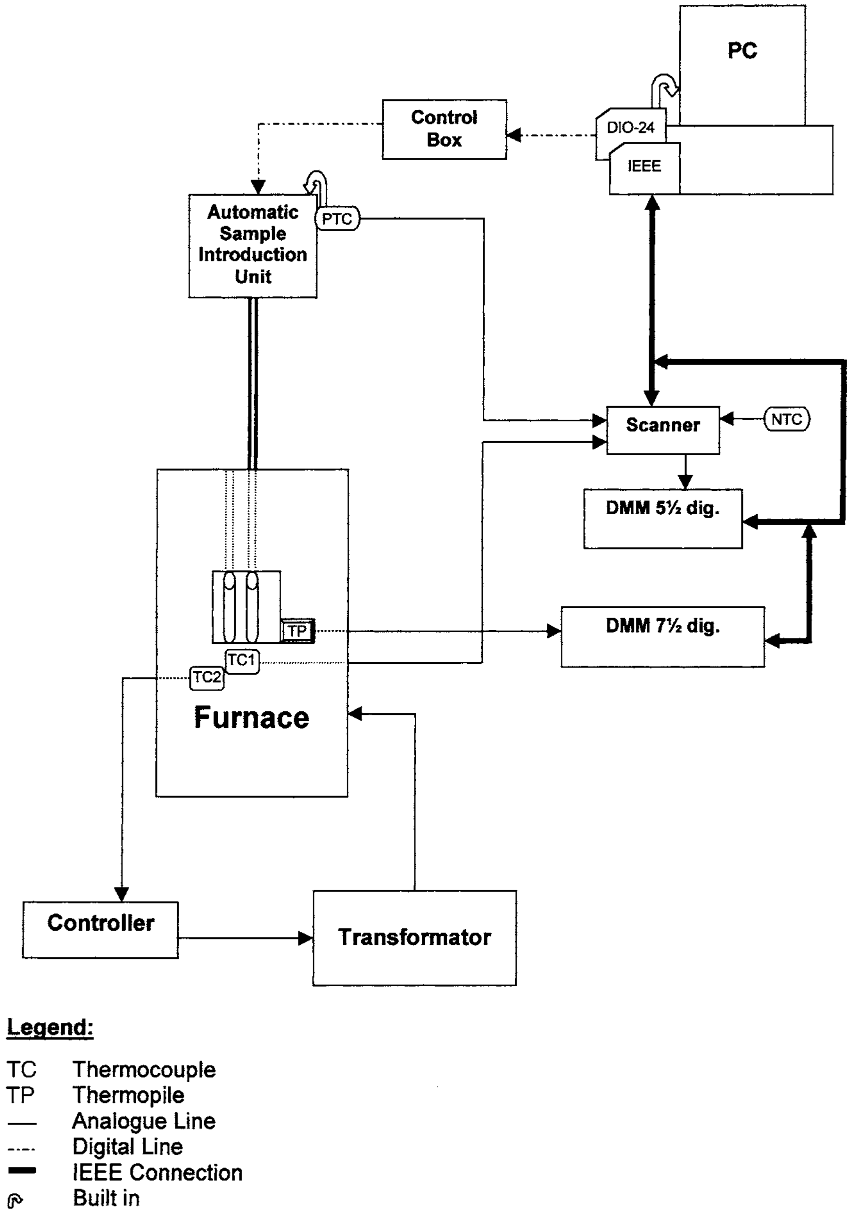

Fig. 1. Schematic diagram of the calorimeter equipment.

Table 1
Commercially available hardware and software

Hardware/Software	Type	Producer
Calorimeter furnace	HT-1000, Calvet 1276	Setaram
Furnace controller	Eurotherm 818	Eurotherm
Furnace transformator	U30	Rusa
Stepping motor	HR23 (four-phase, unipolar, hybrid, 2×3)	Philips
DMM 712-dig	HP3457A	Hewlett-Packard
DMM 512-dig	195A	Keithley
Scanner	705	Keithley
IEEE plug-in board	AT-GPIB/TNT (PnP)	National Instruments
DIO-24 plug-in board	PCI-6503 DAQ	National Instruments
Operation system	Windows98	Microsoft
Control and DAQ	LabView 5.0	National Instruments
Signal evaluation	HiQ 4.0	National Instruments

surrounded by thermopiles (TP) with 420 pairs of thermocouples each. Each TP is mounted on crowns building two concentric cylinders measuring the temperature difference between the inner and outer surface. In this way the EMF of a TP gives a direct information on the heat flow going into or coming out from the calorimeter cell.

The TP are located in the large calorimeter block made of alumina and kanthal which is surrounded by thermal isolating bricks and the electric resistance heating. The signals of the thermopiles can be detected separately but also connected in opposition. In the second case, variations of the temperature of the furnace are self-compensated and temperature differences down to 10^{-5} K can be detected.

The furnace contains two other simple thermocouples. One gives the sample temperature (TC1) and the other one (TC2) is used to control the furnace temperature, which is performed by an Eurotherm 818 Controller.

All thermocouples are made of type S (Pt/Pt–10%Rh). The furnace can be operated from ambient temperature up to 1000 °C. The operation of the Calvet calorimeter is well described in [4].

2.2. Automatic device for the introduction of the samples

Usually one measuring cell of the twin-calorimeter is permanently loaded with a block of about 25 g alumina and serves as the reference cell. The alumina block equalises secondary effects due to thermal conduction of the experimental effects. The second cell is charged with the calorimeter cell, a quartz tube which contains the sample crucible (see Fig. 2, left). This quartz tube is connected to the automatic sample introducing unit (see Fig. 2, middle and right). The entire system is gas-tight and can be evacuated and flushed with the protective gas, i.e. high purity argon.

The automatic sample introducing unit is shown in Fig. 3, top left. It is located in a closeable cylinder-block and consists of a stepping motor which turns around two disks carrying 15 small crucibles each mounted on a small axis. The crucibles are held by the wall of the cylinder but can turn around their proper axis (see Fig. 3, top right). The disks are stacked in a way that by turning the crucibles they reach a position to turn over successively and discharge their load (see Fig. 3, bottom left). So, alternating from the lower and upper disk the specimen put into the small crucibles fall in a funnel and drop to the calorimeter cell into sample crucible. In Fig. 3, bottom right, the two disks with the rings to fix the crucibles for loading and introduction in the sample introducing unit are shown.

For the automation of the addition of the specimen it is necessary to control the stepping motor. For 30 crucibles it takes 1/30 of one complete revolution for a single drop. To turn the disks with the crucibles a unipolar four-phase hybrid stepping motor (Philips) was employed, which performs 200 steps of 1.8° each for one complete revolution. Since this gives not an integral number of steps for a single drop, we programmed two periods of seven and one of six steps for

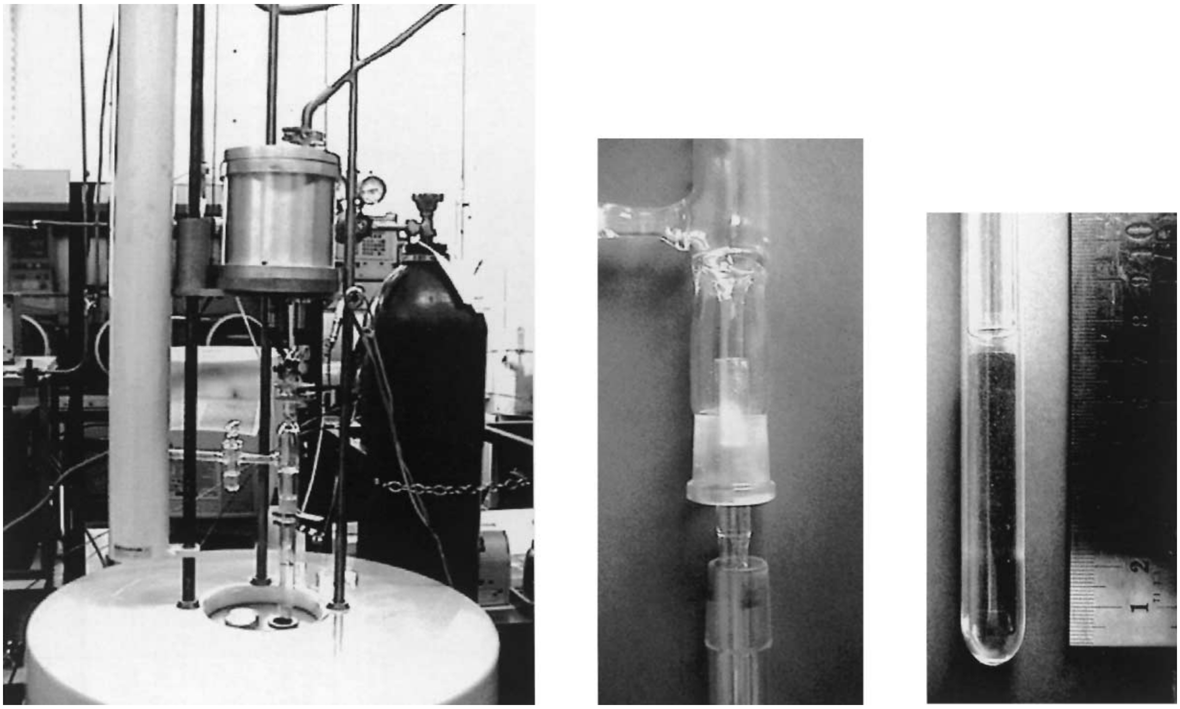

Fig. 2. The automatic sample introduction unit and the connection to the calorimeter furnace.

three drops. Ten such triple-periods add exactly to one complete revolution.

For digital controlling of the stepping motor we constructed an electronic circuit as shown in Fig. 4. The input is given as a binary number with three digits. Two digits are sufficient to control the stepping motor in full step mode. In the sequence 0, 1 $\rightarrow$ 0, 0 $\rightarrow$ 1, 0 $\rightarrow$ 1, 1 $\rightarrow$ 0,1 the motor is turned clockwise, in the reverse sequence it is turned counter clockwise. The third digit controls the current flowing permanently through two of the four coils of the stepper motor. A current of 2 A is necessary to turn the motor. If the third digit is set to 1 the maximum current of 2 A flows. Setting it to 0 a current of 0.2 A is maintained which is sufficient to hold the motor in this position. The full current would heat the entire unit and the samples up to about 40 °C. To avoid these unfavourable conditions, we reduce the current until the next drop.

To gather the digital input signals we use a DIO-24 PCI-card from National Instruments, with 24 digital I/O-lines at three eight-bit ports. It can be easily plugged into the mother board of a PC. The application software we used was Laboratory Virtual Instrument Engineering Workbench (LabView, National Instruments), which is able to read out and write on the digital lines. The bidirectional communication is very important for our application, because the position of the motor is not directly linked with a certain setting of the input signal but changed according to the appropriate sequence of the binary control number. The program has to read the current number in order to change it in the right way to perform the desired action of the motor.

2.3. Heat flow and temperature measurements

In Table 2 the sensors for measuring the signals of heat flow and temperature are listed. The thermopiles combined in opposition are connected to a 71/2-digit digital multimeter from Hewlett-Packard. The instrument is connected to the IEEE-card, which was plugged to the mother board of the PC. The IEEE-card can be finally controlled by LabView using the IEEE commands of the instrument. Each sample addition generates a heat flow which needs about

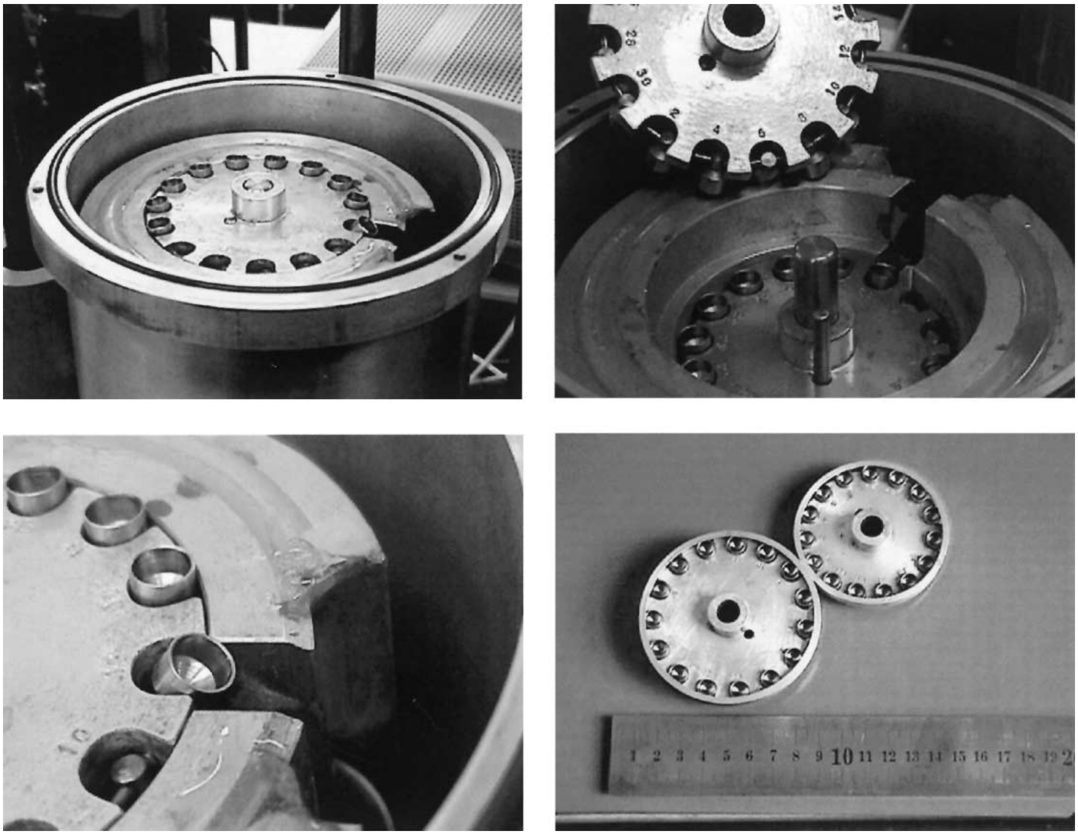

Fig. 3. The automatic sample introduction unit. Some parts and their function.

30 min until thermal equilibrium is reached and the thermoelectric signal is back to the baseline. During this period the signal of the thermopiles, which is proportional to the heat flow, is scanned with a certain sampling rate. The duration of the period, the time of the thermal effect and the sampling rate can be chosen individually. To avoid real time problems with the PC it is preferable to use the timer function of the multimeter instead of the PC. This instrument is able to write up to 1000 values in certain intervals to a buffer, which can be read out by the PC when it is not busy.

The temperature thermocouple and the two resistors are connected to a 51/2-digit digital multimeter via a 10-channel scanner, both from Keithley. The scanner controls the transmission of the signals from the different sensors to the multimeter. Both instruments are connected to the IEEE-card in the PC. Off course, parameters for scanning and measurement can be adjusted via the LabView front panel. The three

Table 2
Sensors for heat flow and temperature measurements

Sensor	Type	Magnitude	Proportional to
Thermopile (TP)	Pt/Pt–10%Rh (420 pairs)	Voltage (V)	Heat flow
Thermocouple (TC1)	Pt/Pt–10%Rh	Voltage (V)	Sample temperature
NTC-resistor	NTC 1000	Resistivity (Ω)	Drop temperature
PTC-resistior	Pt 100	Resistivity (Ω)	Room temperature

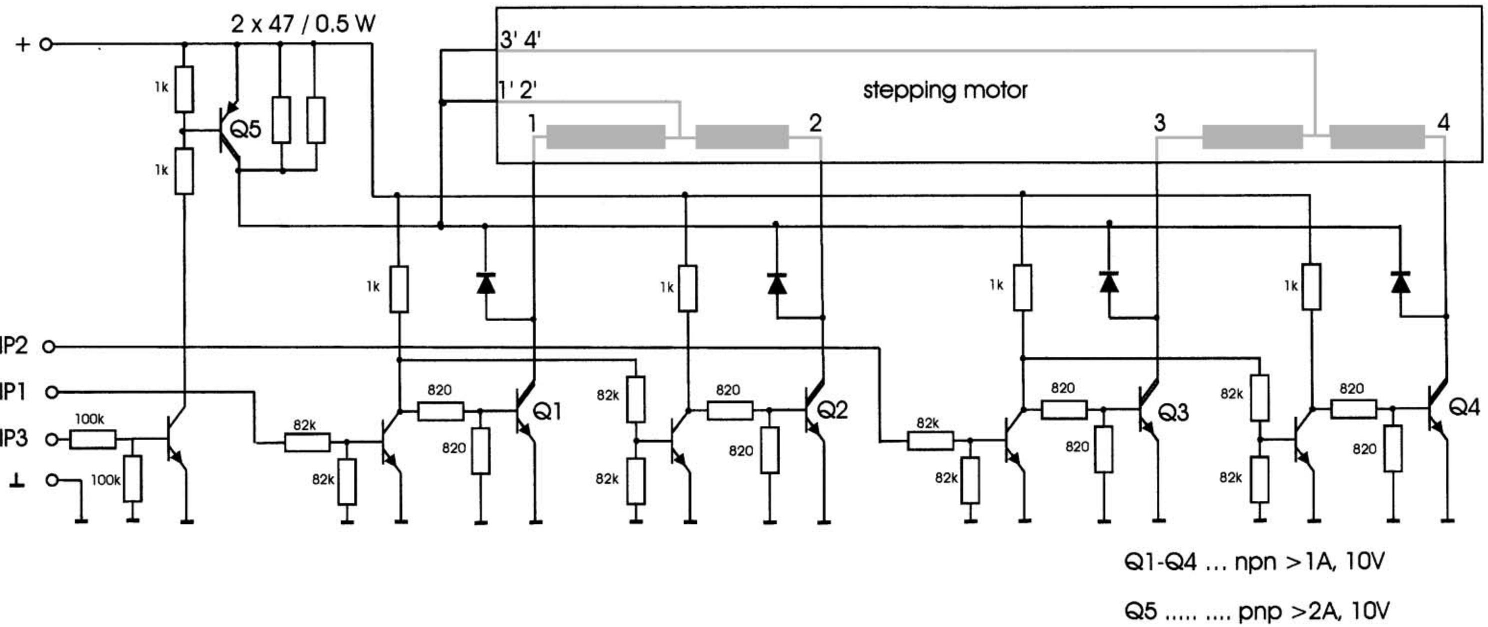

Fig. 4. The electronic circuit of the control box for the stepping motor.

temperature values were scanned before each drop. The room and drop temperature were calculated from the resistivity of the two resistors. The sample temperature was calculated from the EMF of the thermocouple close to the experimental cell in the furnace corrected for the reference junction at the measured room temperature. The calculations were directly performed in LabView and the temperatures were indicated at the front panel of the virtual instrument.

2.4. Programming with LabView

LabView is a development environment based on the graphical programming language G. It uses terminology, icons, and ideas familiar to scientists and engineers and relies on graphical symbols rather than textual language to describe programming actions.

LabView is integrated fully for communication with hardware such as GPIB (IEEE), VXI, PXI, RS-232, RS-485, plug-in data acquisition boards and contains comprehensive libraries for data collection, analysis, presentation and storage.

LabView programs are called "Virtual Instruments" or VI. The VIs can be linked to each other in a certain hierarchy. One VI can serve as a Sub-VI of another VI. A VI instrument consists of a front panel and a block diagram. The front panel is the graphical user interface of the VI. It can contain knobs, push buttons, graphs and other controls and indicators. The block diagram is the graphical source code of the VI. Controls and indicators of the front panel are associated with terminals in the block diagram. In the block diagram you program your VI to control and perform functions on the inputs and outputs you created on the front panel. It can also include functions and structures from the built-in VI libraries and other Sub-VIs. The data flow between the nodes (terminals, functions, structures, Sub-VIs) is programmed by graphical "wiring" in the block diagram. Therefore, the block diagram is also a flow chart of the program represented by the VI. One can set breakpoints, animate program execution to see how the program executes, and single step through the program to make debugging and program development easier.

Acquired data can be exported in different formats in a text file or directly in common software products (i.e. Excel) or evaluation software provided by National Instruments (i.e. HiQ).

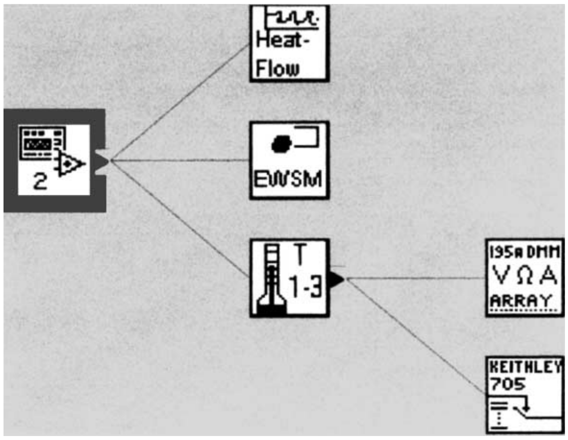

Fig. 5. The hierarchy of the VIs.

For our application we programmed a Main-VI containing three Sub-VI. One serves for control and data collection with the HP3457A multimeter (heat flow), the second one for control of the scanner and the DMM from Keithley (temperature measurements) and the third one for control of the stepping motor. The hierarchy of the VIs is graphically presented in Fig. 5.

These three Sub-VIs are connected and controlled by the Main-VI. It's front panel contains all controls and indicators to enter experimental parameters (period time, drop delay, sampling rate, etc.) and monitor running measurements (number of performed drops, data points per period, etc.). A waveform chart at the front panel of the heat flow Sub-VI indicates graphically the time dependent heat flow of the current experiment (see Fig. 6).

The block diagram of the Main-VI is shown in Fig. 7. One can see the Sub-VIs, their connections and other functions and structures. For more detailed information concerning programming of the VIs described above please contact the corresponding author.

2.5. Data evaluation with HiQ

HiQ from National Instruments is a software for evaluation and presentation of numerical data. Import, calculations, graphical presentations, and the export of data can be also automated by programming HiQ-scripts. The programming language is very similar to MATLAB.

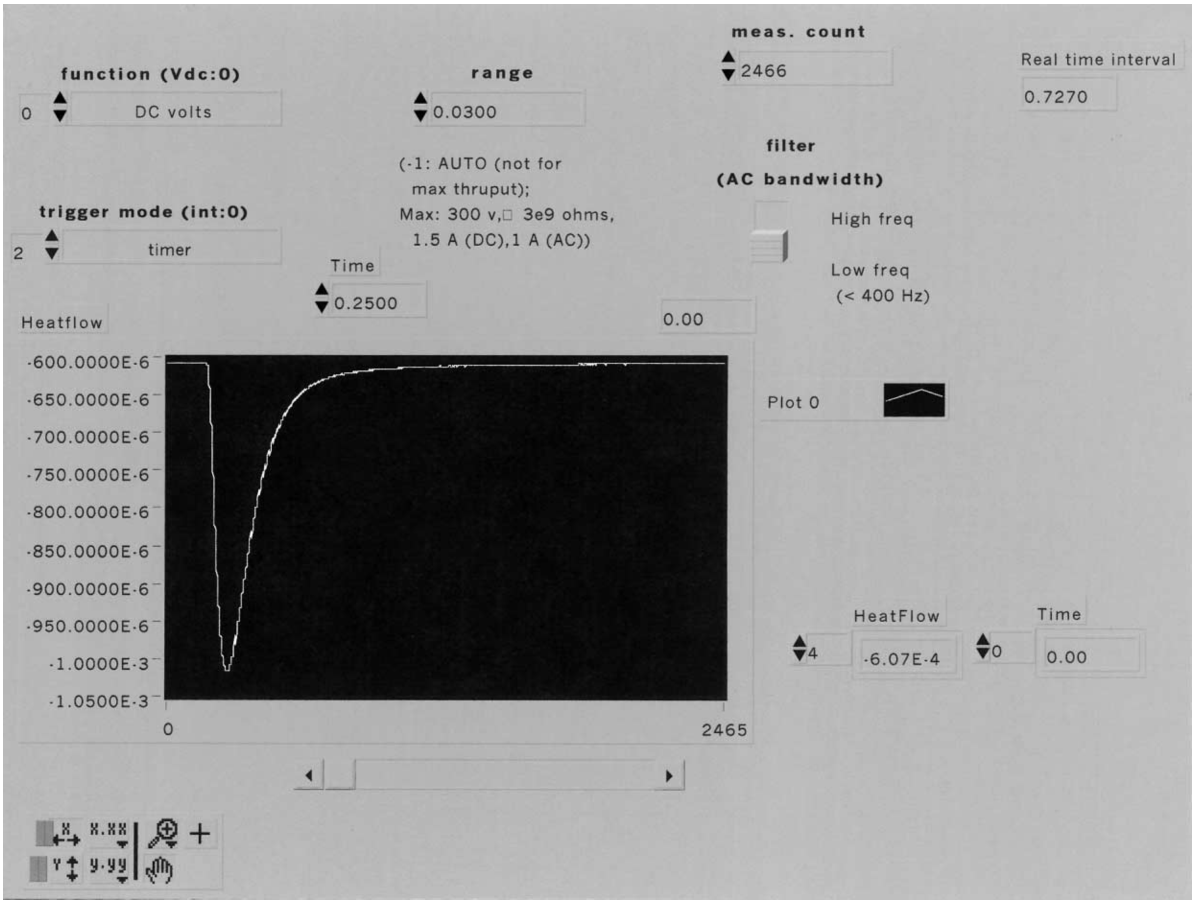

Fig. 6. The front panel of the VI to measure the heat flow rate.

For our applications the data for time and voltage of the thermopiles (proportional to the heat flow) as well as temperature data are written by the LabView VI directly to a HiQ-notebook. The notebook then contains for each period of the experiment a matrix element with two columns (time and voltage) and a text element with the temperatures measured at the begin of the period. A period corresponds to one drop, for more details see above.

In Fig. 8 the HiQ-graph of an evaluated $GaNi_3$ drop (for the determination of C_p-values) is shown. To smooth the signal curve each data point is substituted by the mean value 25 points before and after. The smoothed voltage signal for each period is integrated over the time using a horizontal base line. The base line is determined by calculating the mean of 30 values before the drop. To correct deviations of the horizontal base line from the real signal level after the drop a triangle correction is applied. In that way we know for each point after the drop the value of the integral, if this point would be the upper limit of the integration. This is an advantage for the automation of the evaluation, because the upper limit of the integration (end of the calorimetric effect) can be chosen without changing the integration procedure. In case of considerable changes of the base line or jumps of the base line during the drop period other well described methods of evaluation have to be applied. For the determination of the calorimeter constant (calibration of the heat flow) usually drops of NIST-Al_2O_3 are performed.

Calculations and diagrams as well as export of integrated signal surfaces are made automatically by the scripts we programmed in HiQ. Nevertheless, one has to check very carefully for each signal if the

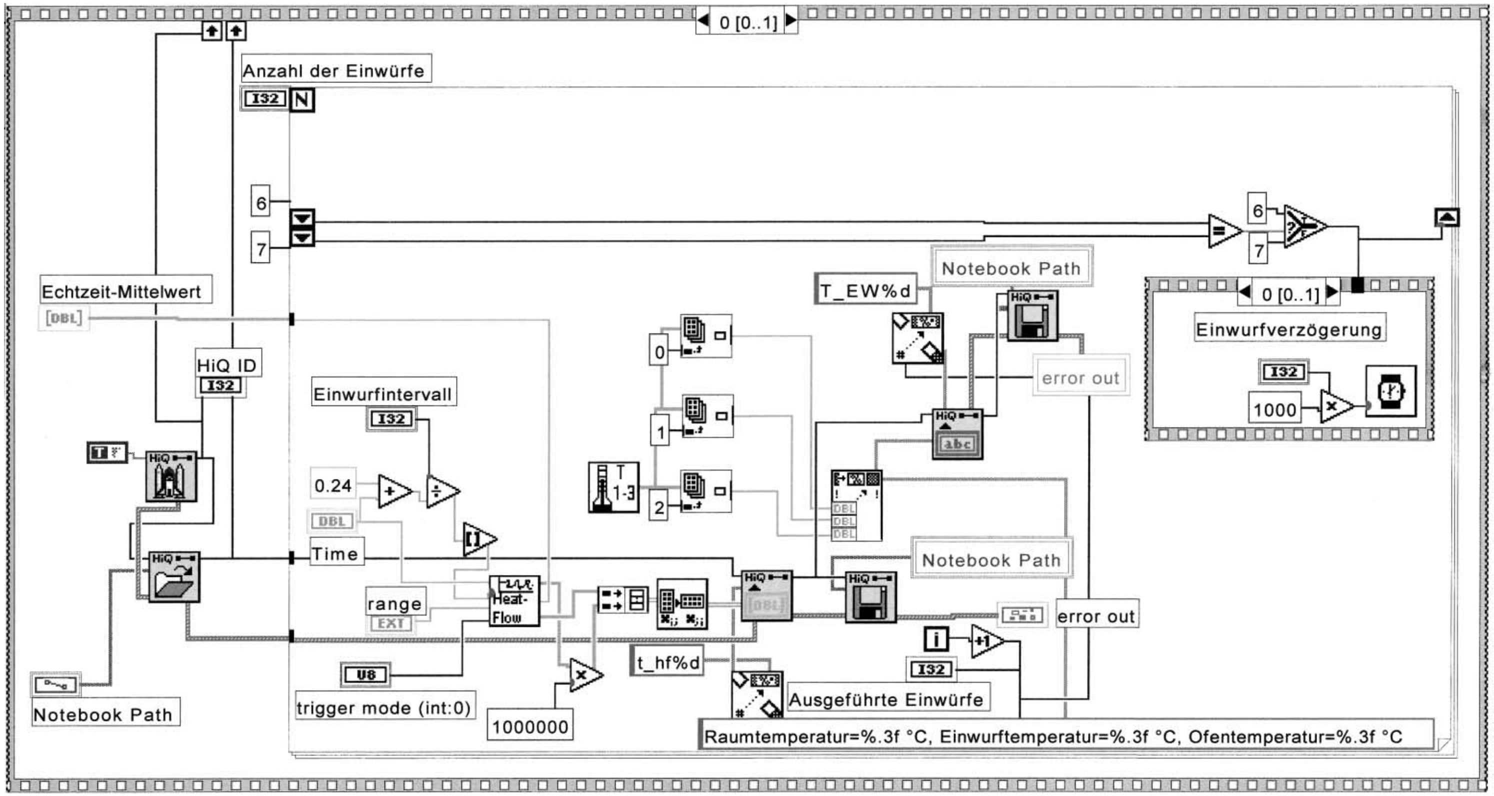

Fig. 7. Block diagram of the Main-VI.

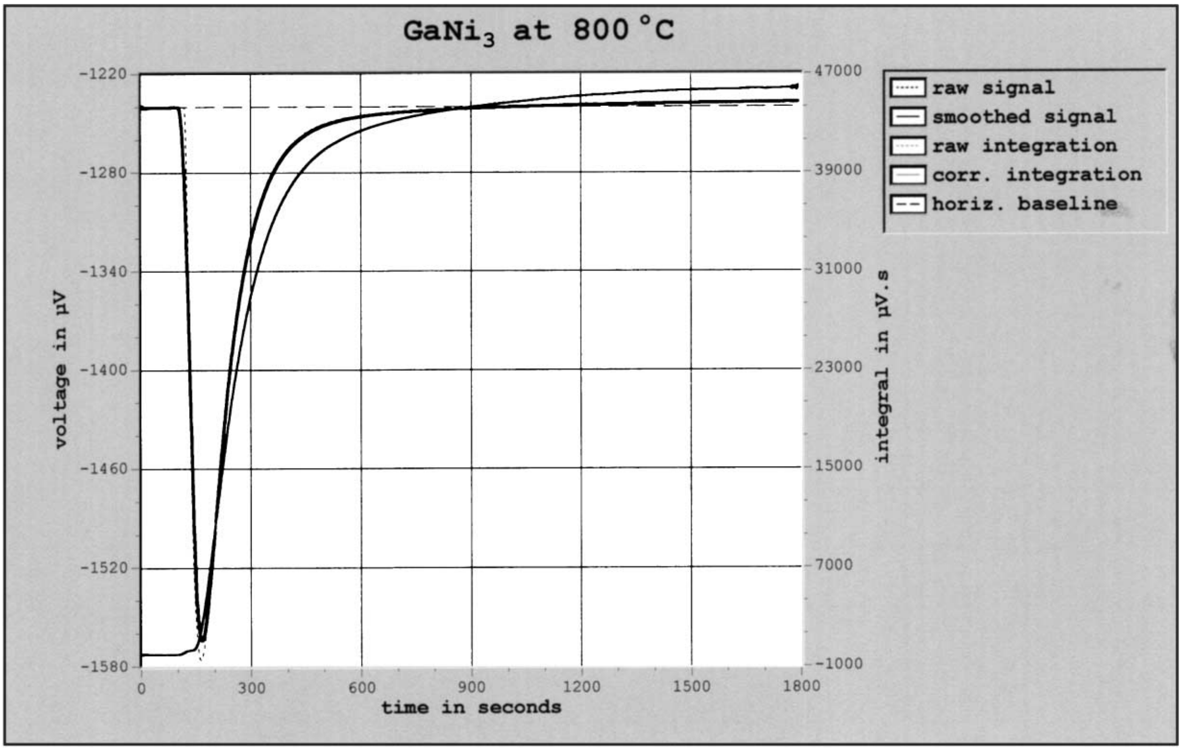

Fig. 8. The 2D-graph of an evaluated drop signal ($GaNi_3$) created in HiQ.

chosen standard procedures and settings are correct. The automation saves time for repeating evaluation steps but can not replace critical examination of the experiments.

3. Results

The Calvet calorimeter is now running measurement series by 24 specimen additions to a starting metal or alloy followed by six calibration drops, usually. As calibration materials we employed α-alumina by NIST. If the thermal equilibrium is reached between 30 and 50 min, which is a reasonable value for liquid alloys formed by the addition of a solid constituent to a liquid metal or alloy, a period of about 60 min will show a reasonable thermogramm to calculate the heat of reaction and to derive the corresponding partial and integral enthalpies of mixing. Therefore, the energetic of a quasibinary section of an alloy system can be determined within 2 days with this operating system. The calibration of the cell, however, has to be determined for each single measurement series in order to take into account the position of the calorimeter cell toward the thermopiles, the different masses, crucible materials and other effects.

4. Conclusions

A low cost up-to-date solution is presented for the control and data acquisition of a high temperature calorimeter. Three already existing instruments (2 DMM, Scanner) and the controller of a automatic drop device have been integrated. The central control unit, where all devices are controlled and all data collected was a Pentium-PC. LabView, the software used, gave us the possibility to create the desired programs.

If you use existing instruments as we did you can get already programmed VI to control them via LabView from National Instruments or directly from the instrument's producer. The offered VIs, however, were adapted for the special application. We made the experience that some of them were not very carefully programmed and contained some errors. Although the authors were not experienced in programming, we succeeded to create appropriate VI for our applications.

Acknowledgements

We gratefully acknowledge that National Instruments offered plenty of possibilities to get support for installation and application of their hardware and software. There were technical support via telephone or Internet, user networks, example data base, customer meetings and training available.

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