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MICROCOMPUTER CONTROLLED EXPERIMENTATION IN CALORIMETRY. APPLICATION TO THE DETERMINATION OF THE PARTIAL ENTHALPIES OF 3*d* TRANSITION METALS IN LIQUID ALUMINIUM

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ABSTRACT

This paper describes the development of a total automation and cheap data acquisition system for a high temperature Calvet calorimeter or an isoperibolic calorimeter with a Commodore 3032 microcomputer. The system has been developed successfully to drive a two-phase stepper motor, to provide programmed control of a 181 Keithley nanovoltmeter and to calculate the total heat effect.

The calorimetric precision for dissolution of 3*d* transition metals in liquid aluminium at 1000 K is about +0.1%.

INTRODUCTION

The calorimeters used were a high temperature Calvet and an isoperibolic calorimeter described elsewhere [1].

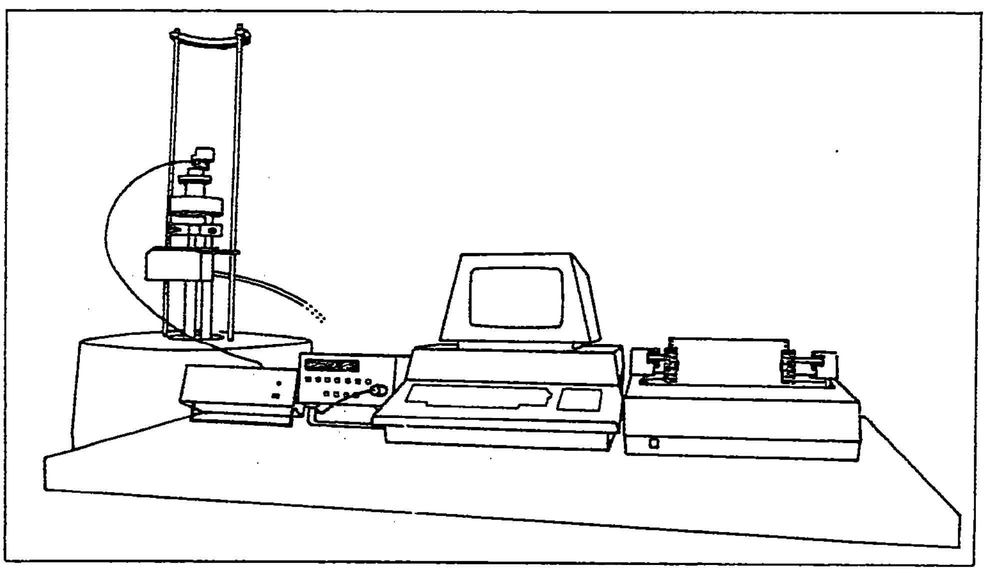

Fig. 1. Data acquisition system.

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The typical period of our experiments is of the order of one hour. The calibration of a calorimeter requires about ten runs for the determination of a heat of dissolution.

The repetitive nature of the addition of the samples permits the use of automatic control, thus eliminating human intervention which could disturb the temperature control of the room. This paper describes the programming of a stepper motor driving an injector and data acquisition from a 181 Keithley nanovoltmeter. Moreover, each thermal effect is recorded by the microcomputer. This system, shown in Fig. 1, has been used to determine the partial enthalpies of 3*d* transition metals for infinite dilution in aluminium at 1000 K.

SELF-ACTING INJECTOR

The self-acting injector (Fig. 2) is an enclosed cylinder having 23 small spouts. A stepper motor turns the enclosed cylinder and drives successively

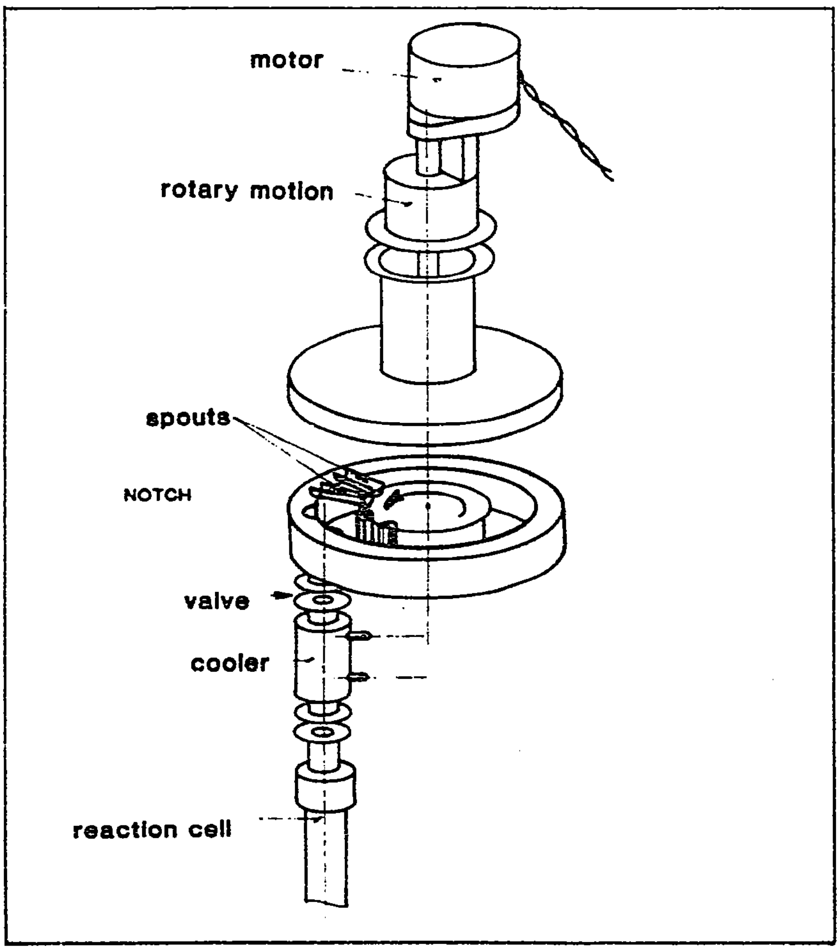

Fig. 2. The self-acting injector.

the spouts towards a notch. When a spout reaches the notch, it rocks and lets the sample drop into the reaction cell.

INTERFACING

IEEE 488 bus

The Commodore 3032 uses the IEEE 488 standard interface bus, already well known. There are presently three devices connected to the bus in our acquisition system: a teleprinter (Commodore 3022) and two digital voltmeters (Keithley 181).

Parallel user port

There is a second port brought out from the CBM main logic board and called the parallel user port. It consists principally of 8 individually programmable bi-directional input/output lines: PAØ 7. These are programmed by initialisation of decimal address 59459 using POKE basic instruction. The command of the output register is located at decimal address 59457 and similarly programmed.

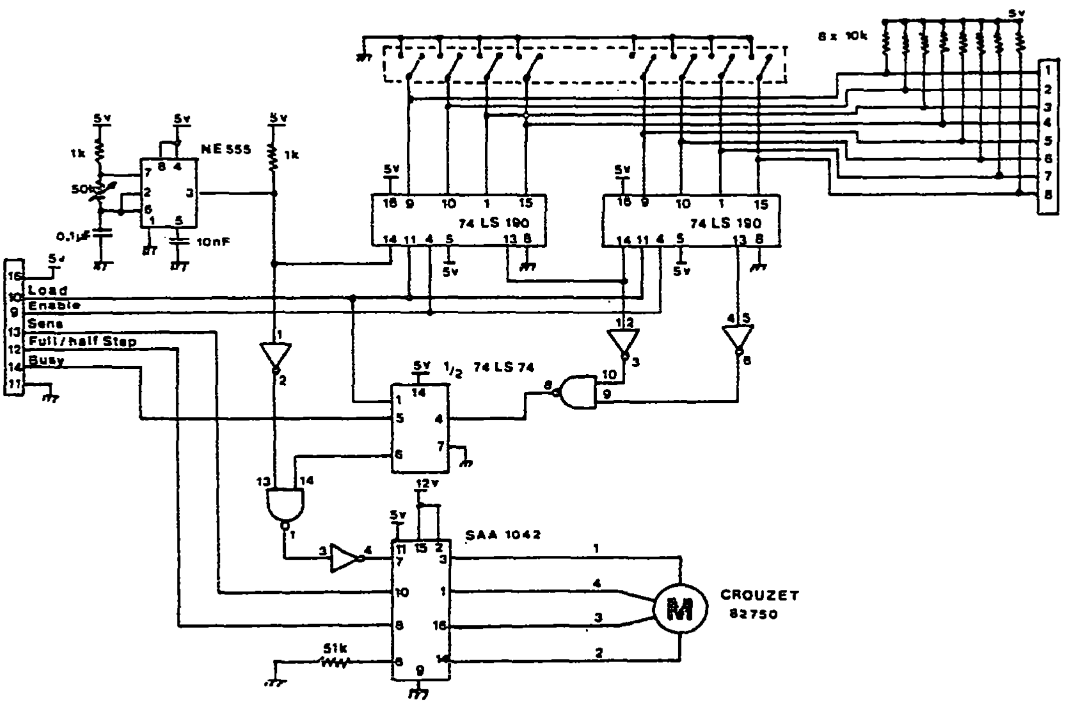

Fig. 3. The stepper motor drive circuit.

STEPPER MOTOR DRIVE DEVICE

Circuit

This is shown in Fig. 3. A strobe pulse is generated by a frequency oscillator NE 555. Associated resistors and capacitors are chosen to suit the rotation speed of the stepping motor. Two 4-bit counters connected together are loaded with the data transmitted in a bit-fashion on 8 lines which can either be brought out from the controller and programmed by it or fixed manually by an 8-switch system. The two counters are associated with a NAND gate and a D flip-flop 74L74 to provide the appropriate gating signal. This signal is sent to the two-phase stepper motor drive SAA 1042 chip from Motorola. Two other lines coming from the controller, connected to the SAA 1042, command the rotation direction and the half- step function.

Programming

The system is shown in Fig. 4. The ten programmable lines of the user port of the CBM 3032 are not sufficient to load the counter with data programmed by the computer and the switch system must be used. Five lines (PAØ → 4) are used: 4 (PAØ → 3) in the OUTPUT mode, one (PA 4) in the INPUT mode. The output register is then used to initiate the circuit and

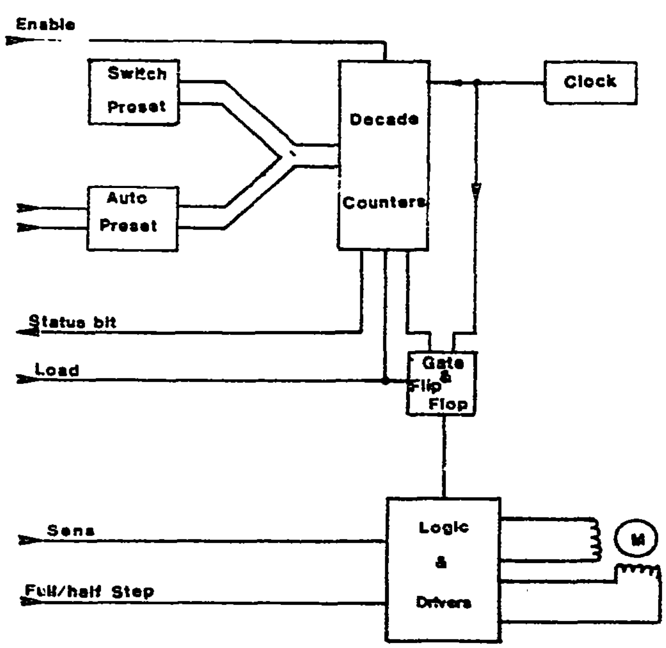

Fig. 4. The stepper motor drive logic connections.

functions as follows
PAØ: LOAD, bit = 1
PA1: Half-step, bit = 1
PA2: Direction, bit = 1
PA3: ENABLE, bit = Ø
and to command each step by one pulse on the LOAD line: bit of PAØ to Ø, then back to 1.

The input line is used to check the status bit of the motor interface: PA4, bit = 1 if busy using PEEK basic instruction.

Step advance subroutine program

```
1Ø POKE 59459,15
2Ø POKE 59457,7
3Ø POKE 59457,6
4Ø POKE 59457,7
5Ø A = PEEK (59457)
6Ø IF A 24Ø THEN 5Ø
```

SOFTWARE

The system software is divided into two sections: an acquisition section and an exploitation section.

The acquisition section provides for programming of the 181 Keithley nanovoltmeter via the bus IEEE and of the two-phase stepper motor via the user port. The exploitation section calculates the total heat effect when all data are recorded and stored by the microcomputer. The mean values of the first and last ten measurements are used as, respectively, initial and final base line values, the end of the experiment being determinated by calculation of the curve tangent. Under ideal conditions, the initial and final base lines have the same values and the surface of the thermogram can be approximated by a series of trapezes of the same width. Under practical conditions, however, this may not be achieved and a new base line is obtained by linear interpolation of the initial and final base lines. In some cases, to obtain a greater precision, the final portion of the integral can also be computed by calculating the time constant of the decaying curve over a series of points and the computer extrapolates the curve to infinity.

RESULTS

The partial enthalpies of some transition metals at infinite dilution in aluminium at 1000 K have been obtained using the previously described

TABLE I

Experimental data of calorimetric determination

Element	Ref.	T(K)	ΔH^{∞} (kJ)	σ (kJ)
Ni	This work	1000	−137.30	0.85
	2	948	−139.89	2
	3	1173	−128.85	
	4	1023	−139.20	
	5	1100	−136.50	1.5
Co [a]	This work	1000	−138.5	0.85
	5	1100	−140.5	1.5
Mn [b]	This work	1000	−81.1	0.85

[a] Referred to Co β

[b] Referred to Mn β

method and calorimeter [1]. The experimental results are shown in Table 1.

Comparison of our experimental values and earlier published results shows a good agreement.

In conclusion, we can say that the time taken to set up the system is extremely short due to the modular nature of the hardware and the ease of program editing in the computer. This system has been in operation for several months and the precision of our calorimetric results has been greatly increased.

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