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Temperature Control Programs for Precision Water Bath in Heat Exchange Calorimeter

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1. Introduction

In the heat exchange calorimetry,[1] sample and reference vessels were fixed differentially in a thermostatted water bath. Heat evolved in the sample vessel was exchanged freely with the ambient water. The thermal behavior was expressed by a simple differential equation. Temperature in each vessel was detected by thermistors used as a thermal sensor. Total heat and heat evolving rate owing to chemical reactions or electrical heating were estimated via analog and digital computations of observed signals of temperature.

The temperature control of bath water may be indispensable for precise and accurate estimation of smaller heat effect, as the calorimeter was usually handled in a non-airconditioned laboratory. The thermal fluctuation of bath water must be kept within the range required from minimum heat amount to be measured. In addition, the temperature drift may be also limited to a narrow range according to resistance vs. temperature characteristic of a set of thermistors used in each vessel.

In the present report, simple temperature control system of precision water bath was developed for practical use. Cooling water lower than the set temperature was circulated in the bath, and the heater current was controlled for the bath water to keep the set temperature by P I D (proportional, integral, and derivative, respectively) control technique[2] modified by the authors. The temperature deviation, which is the difference between the current temperature and the set temperature, was observed periodically by a thermistor and acquired into a microcomputer via AD (analog-to-digital) converter. After calculations of control input by presented programs, control signals were sent to DA (digital-to-analog) converter and then to the heater circuit. The hardware was made as simple as possible and fine temperature controls were carried out by the software. The dynamic range was selected automatically in the program, so that the relation of input range to output may be optimized. In the report,

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programs for automatic control were presented in flow diagrams. Details of the hardware and controlled results were omitted.

2. Experimental

Apparatus The temperature control system set-up in the work is shown in Fig. 1. A thermistor to measure temperature of the bath water, screws of a motor-driven stirrer, glass spiral tubing for cooling water, and a heater element were included in the bath. Sample and reference vessels, and a titrant reservoir for thermal equilibrium were omitted in the figure for simplicity.

Procedure Sample and reference vessels for calorimetric measurement were set in the water bath of the calorimeter. Titrant reservoir was also set for thermal equilibrium. The bath was filled with water near the set temperature. Circulation of the cooling water was started and then control programs executed. The bath temperature was automatically maintained at the set temperature.

3. Results and Discussion

Ten control deviations T including the latest acquired data were used for calculating output terms of P, I, and D. The latest data are written as $T(0)$, and n in $T(n)$ represents T acquired before n sec. Constants of $K1$, $K2$, and $K3$ are coefficients of P, I, and D, respectively, and selected from progress of t, which were given by trial and error method. Output parameter H to be sent to the heater element via DAC was finally calculated as summation of P, I, and D, where $P=T(0)\times K1$, $I=M/K2$, and $D=(T(0)-T(1))\times K3$, and M is an integrated value of control deviation.

Simplified flow chart of programs is shown in Fig. 2. Calculation routines of P, I, and D are shown in Figs. 3-7. All programs were written in BASIC and amounted to 8 kbyte.

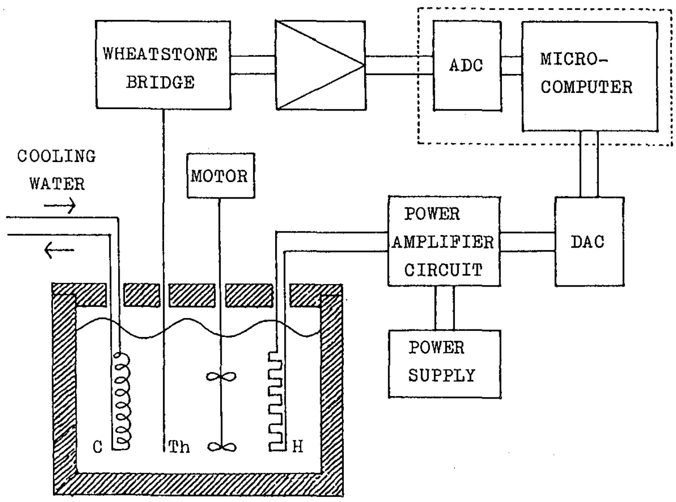

Fig. 1. Schematic diagram of the whole control system.
Th, Thermistor; H, Heater; C, Cooler.

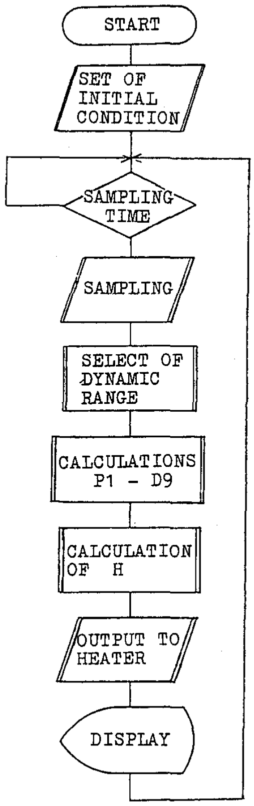

Fig. 2. Simplified flow chart of programs.

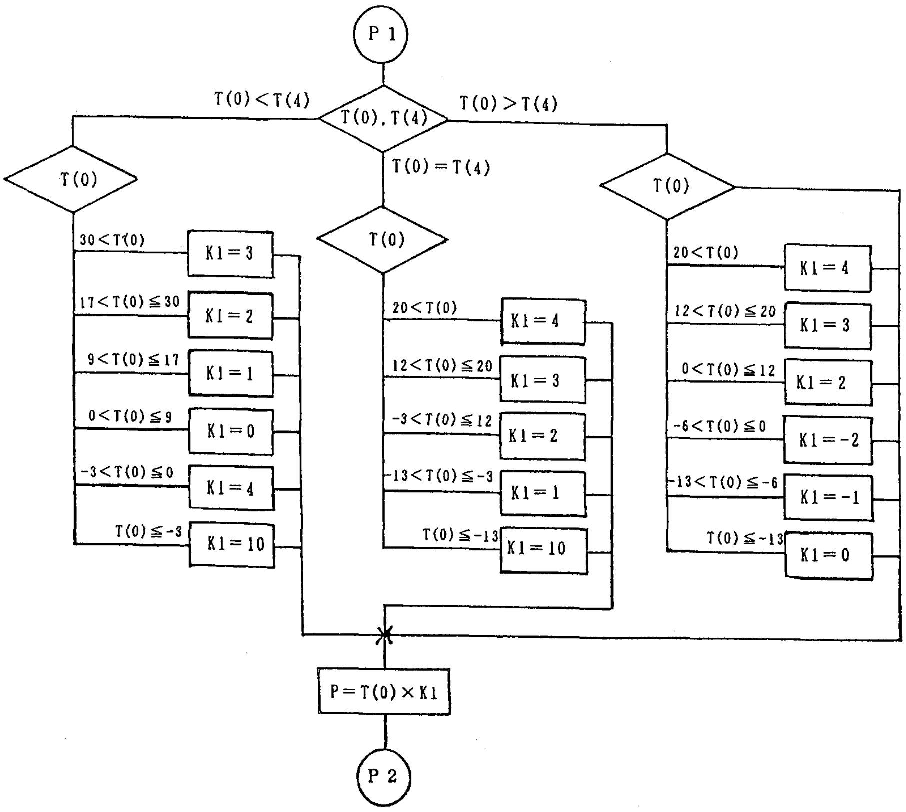

Fig. 3. Flow chart calculating proportional control term

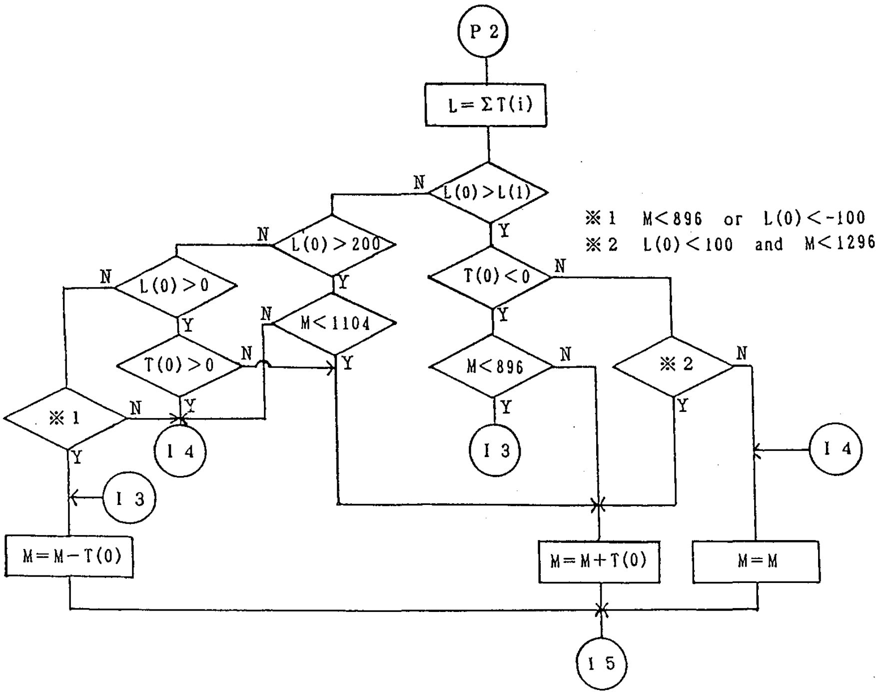

Fig. 4. Flow chart calculating integral control term. Part 1.

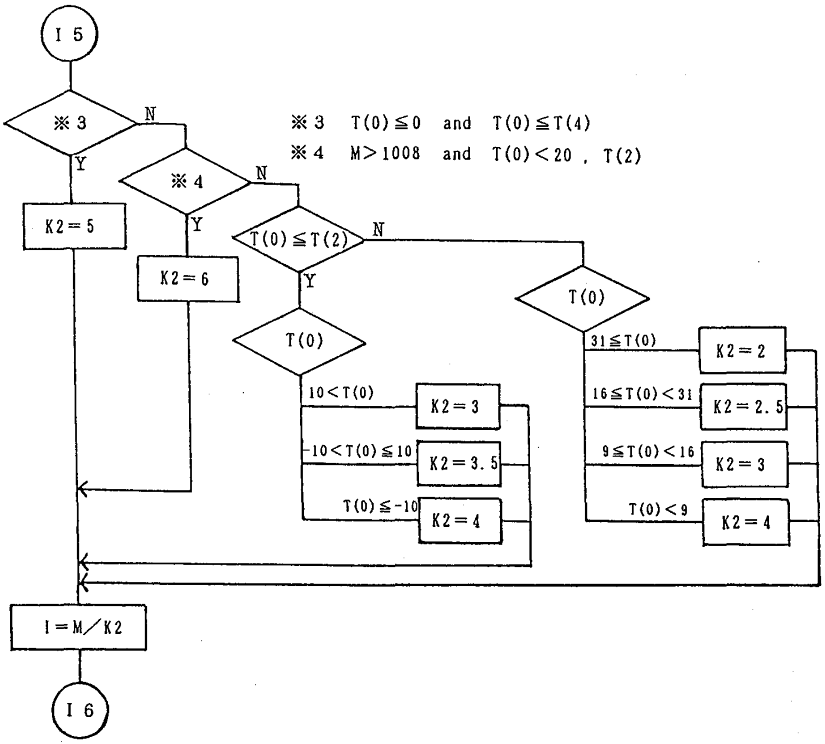

Fig. 5. Flow chart calculating integral control term. Part 2.

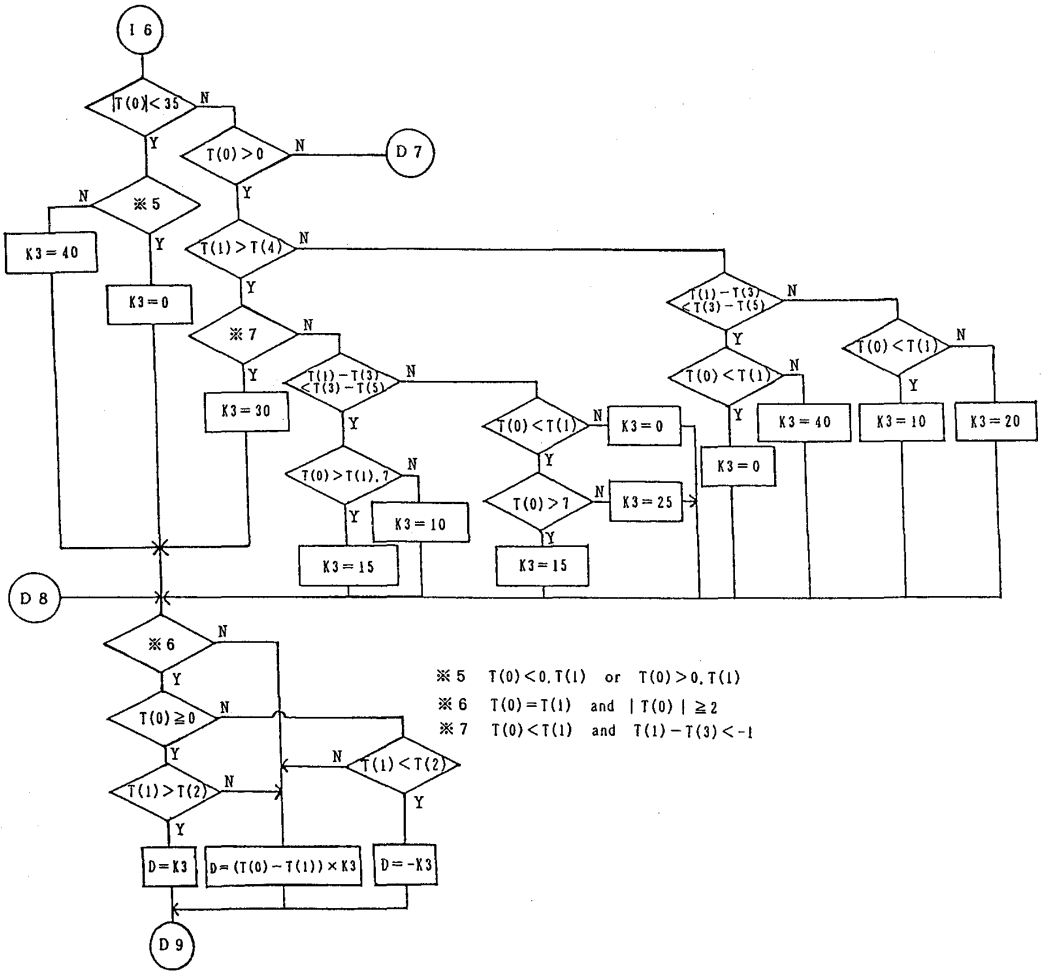

Fig. 6. Flow chart calculating derivative control term. Part 1.

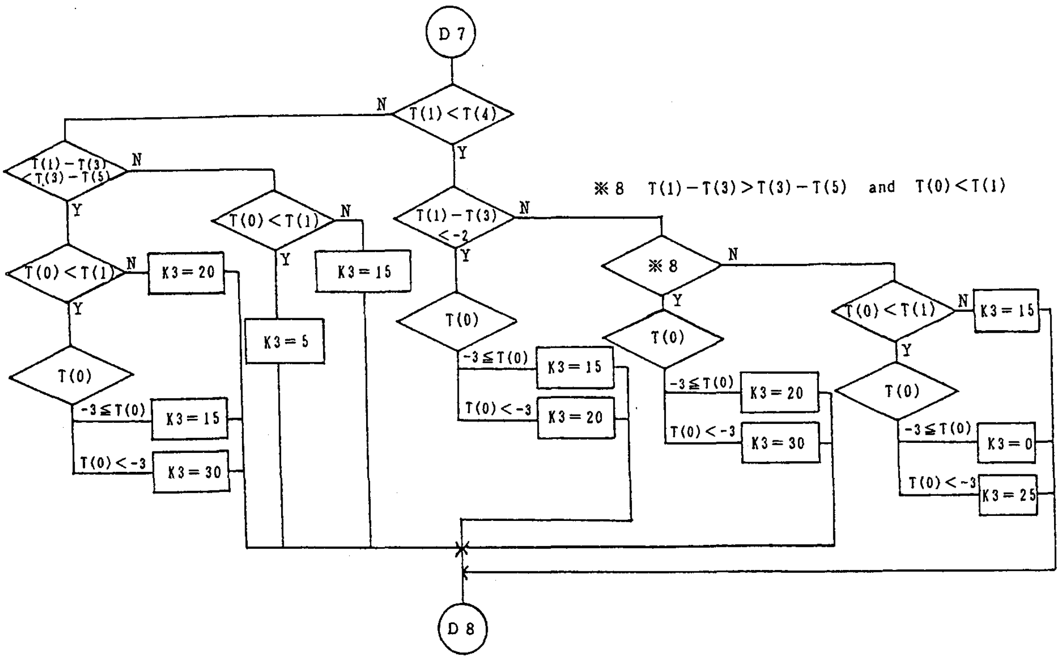

Fig. 7. Flow chart calculating derivative control term. Part 2.

Calculated output parameter or control input H of 1000, instead of 1024, was correlated with 10 V in full scale of control value.

Summation M of control deviation was used in I to prevent the bath temperature from stabilizing at an off-set temperature.

When temperature deviation, which was observed as unbalanced voltage of a Wheatstone bridge including the thermistor, was large at starting period of control, sometimes output I did not work effectively for restoration of drift due to sudden increase or decrease of M. Same behavior may be found when M is over an upper limit, provided that the temperature of bath water drifted. M was replaced by a value less than the upper limit to make speedy response according to T.

Output D was a parameter to deal with sudden change in temperature. When the temperature is far from the set value, $K3$ was given only from comparison of $T(0)$ and $T(1)$. However, as the temperature approached gradually to the set value, progress of $T(3)$ and $T(5)$ which reflected the direction of tangent and extent of the difference was also taken into consideration to decide $K3$. If there was no change in the temperature, $K3$, instead of zero, was given to D.

The proposed program was useful in practice. However, since most of constants were selected by trial and error method, the program was very complicated. More sophisticated and general control system should be developed for the calorimeter.

References

[1] M. Nakanishi and S. Fujieda: Anal. Chem., **44**, 574 (1972).
[2] S. Samukawa: Transistor Gijutsu, **1984**, 459 (in Japanese).