

HIGH PRECISION AUTOMATIC CAPILLARY VISCOMETER*

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ABSTRACT

An automatic capillary viscometer with optical fibres as cold light source and a thermostat in which the temperature can be regulated within $\pm 5 \times 10^{-4}^{\circ}\text{C}$ have been developed for the determination of flow time with an accuracy of $\pm 4 \times 10^{-3}$ sec. The basic principle, construction, specifications as well as the main features of this instrument are described.

INTRODUCTION

Capillary viscometer is very useful for measuring viscosity, molecular weight, branching and other related characteristic parameters of polymers. It has been used extensively for its reliable theoretical basis, simplicity of the configuration, reasonable accuracy, ease and convenience for operation. Some articles on automatic viscometer have been published since 1970^[1-9]. In this paper, an automatic capillary viscometer with optical fibres as cold light source is reported. The fluctuations of solvent flow time in this viscometer are less than $\pm 4 \times 10^{-3}$ sec, and the accuracy of bath temperature is within $\pm 5 \times 10^{-4}^{\circ}\text{C}$.

1. The principle of automatic time measurement

The automatic time measurement with the capillary viscometer is based on the principle of photoelectric effect. Photoelectric transducers are mounted above and below the measuring bulb. As the refractive index of air is different from that of liquids, the light intensity absorbed by the phototriode is different and produces different photocurrents, which could switch on or off the quartz crystal controlled counter (see Fig. 1).

When the liquid fills up the viscometer, because of focalization, the phototriode is switched on by the strengthened light signal received. When the liquid level goes down, the incident light is reflected by the meniscus and the phototriode is switched off by the weakened light signal received.

The schematic circuit diagram of the automatic photoelectric time measuring system is shown in Fig. 2. If the phototriode (3DU 12) is on, the base potential of BG_1 will be high, BG_1 will be on and so will be BG_2 ; meanwhile the base potential of BG_3 will be low, BG_3 will

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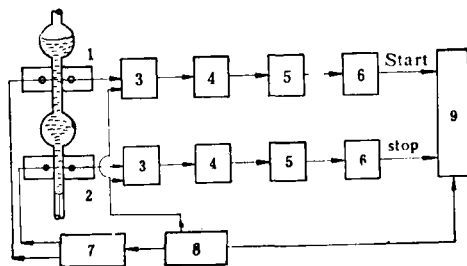


Fig. 1 Block diagram of the automatic photoelectric time measuring system

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|------------------------------|----------------------------|---------------------------------|
| 1. Upper input; | 4. Inverted amplification; | 7. Cold light source; |
| 2. Lower input; | 5. Wave reshaping; | 8. Power supply; |
| 3. Impedance transformation; | 6. Pulse output; | 9. Digital frequency indicator. |

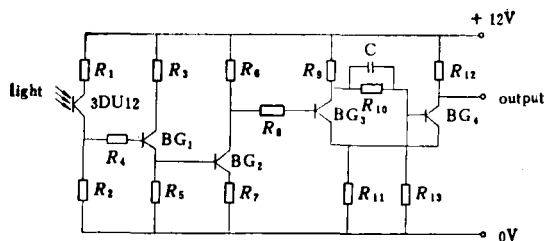


Fig. 2 Circuit schematic diagram of the automatic photoelectric time measuring system

be off; BG_4 will be on and a low potential output will be given. On the contrary, if the phototriode is off, a high potential output will be given. When the output signals are sent to the "start" or "stop" of the counter, automatic time measuring can be realized.

2. Details of construction

This device is comprised of an automatic capillary viscometer, a precision thermostat and other accessories (see Fig. 3).

The automatic capillary viscometer includes a modified viscometer of the Ubbelohde type, a cold light source, photoelectric transducers, a pulse counter and a viscometer regulating support.

The modified Ubbelohde viscometer is shown schematically in Fig. 4. Its most important parameters are as follows: the diameter of the capillary $d = 0.35$ mm, the length of the capillary $l = 170$ mm, the minimum volume needed to start the experiment $v = 2$ ml.

The flow time of pure THF at 25°C is about 170 sec. The capillary is designed in a single spiral form and bent smoothly without any dead region, so that it can be located in the centre of the well-distributed temperature field, the height difference between the entrance and the exit can be decreased, and a fully developed velocity distribution of the fluid in laminar flow can be obtained.

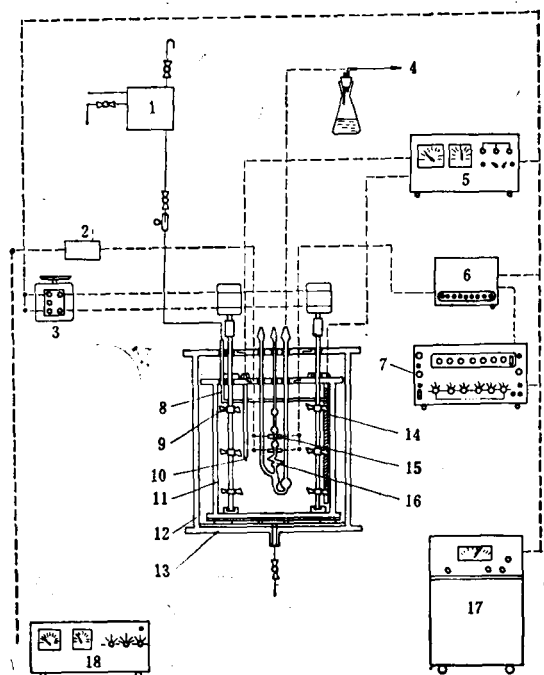


Fig. 3 Scheme of the automatic viscometer device

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|---|---------------------------------|
| 1. High potential energy vessel of cooling water; | 10. Thermistor; |
| 2. Cold light source; | 11. Inner thermostat container; |
| 3. Auto-transformer; | 12. Outer thermostat container; |
| 4. To vacuum system; | 13. Location support; |
| 5. Precision temperature automatic controller; | 14. Trimming heater; |
| 6. Pulse generator; | 15. Photoelectric transducer; |
| 7. Digital frequency indicator; | 16. Capillary viscometer; |
| 8. Cooling pipe; | 17. A.C. voltage stabilizer; |
| 9. Stirrer; | 18. D.C. voltage stabilizer. |

The precision thermostat includes an outer cylindrical vessel and an inner one, a temperature measurement system and a temperature control system, a preheater and a trimming heater, two sets of stirrers, a flow rate regulation system of cooling water.

The temperature control system composed of a high sensitive Wheatstone bridge, a microvolt amplifier, PID regulator, silicon controlled trigger and electric heaters (see Fig. 5).

3. Basic design features

The basic design features of this device are as follows:

A. A triple isothermal system consisting of an inner water bath, an outer water bath and the room.

B. An approximate stepless heat compensation method for controlling the temperature with fine adjustment and direct heating.

C. The heating capacity of this thermostat is large. The heating capacity of the inner water bath is 50 kcal/°C, and that of the outer water bath is 25 kcal/°C.

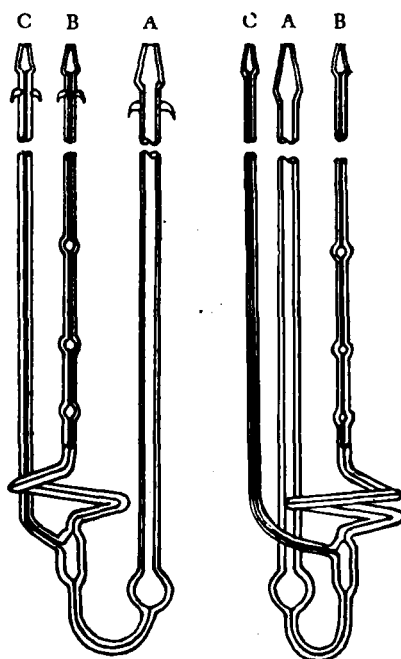


Fig. 4 Diagram of the modified capillary Ubbelohde viscometer (front view and side view)

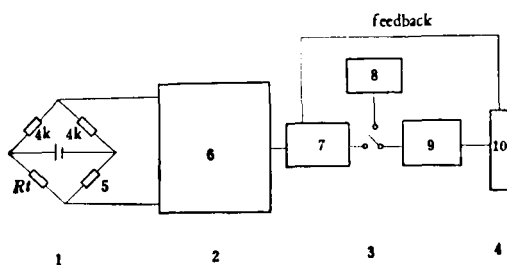


Fig. 5 Block diagram for controlling the constant temperature system

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|---------------------------------|--------------------------------|
| 1. Signal generator; | 6. Microvolt amplifier; |
| 2. Amplification and shift; | 7. PID regulator; |
| 3. Heating voltage controlling; | 8. Manual signal; |
| 4. Heating; | 9. Silicon controlled trigger; |
| 5. Precision resistor; | 10. Heater. |

D. A high efficient stirrer is designed.

E. Optical fibres are used as the cold light source.

F. A modified capillary Ubbelohde viscometer and its automatic time measuring system are designed.

G. Some strict waterproof measures are adopted.

H. Some suitable materials are selected to construct this device.

I. In order to stabilize the pressure and flow rate of cooling water, some measures are adopted.

J. A procedure of cleaning the viscometer is used.

Temperature has obvious influence on viscosity. As temperature varies 0.01°C , the flow time will vary 0.01 sec. In order to ensure the thermostat temperature constant within a long period of time, some efficient measures are adopted. Besides, the influence of viscometer system on viscosity must also be taken into consideration. For this reason, optical fibres are used as the cold light source of photoelectric transducer, and the accuracy for measuring the flow time has been increased.

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