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Improved optical chamber for small measuring cells with temperature control

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An improved optical chamber is described containing a Chatelain prism with controlled heating and temperature control. It consists of placing the prism between two copper plates heated through a miniaturized device that creates a hot air current inside a small acrylic chamber. The heater is a power transistor controlled by an operational amplifier that creates a 3 h ramp generated digitally through firmware in a microcontroller. The cell temperature, obtained through a thermistor, can be controlled to ±0.1 K. © *2005 American Institute of Physics.* [DOI: 10.1063/1.2083167]

The measurement of optical properties over the whole range of a mesophase in liquid crystalline materials is of great importance because it provides valuable information on the orientation and distribution of the component molecules. This is not a simple problem, because it requires a system that will enable heating the sample in a transparent device, the temperature of which must be carefully controlled.

Commercial instruments are adequate when only the transition temperatures need to be known. They are usually combined with microscopes that allow one to visualize a solid to liquid transition at a specific temperature. This is the case of work by Riccó and Dalacanale[1] on macrocyclic columnar liquid crystals, using a Leitz-Panpoht microscope equipped with a Mettler FP-82 heating stage. Also, Mita and Kondo[2] identified transitions in lyotropic phases using an Olympus polarizing microscope with a Mettler PF-5 heating stage.

But, as mentioned above, when the whole mesophase has to be examined the problem becomes difficult to solve because both heating and temperature control must be achieved in small cells that contain thin films of sample, where a thermometer cannot be introduced.

When very small thermistors became available, Seppen[3] made an interesting attempt with a two-stage double-shielded heating system to study the influence of magnetic fields on phase transitions and fluctuations in biaxial liquid crystals. The procedure required the use of two Wheatstone bridges, each with a thermistor, in order to have an adequate temperature control in the sample containing cell.

However, in all the cited cases the heating and temperature control in small cells implied in general a surrounding of the cell completely with some kind of heating system. It could be water circulation, as in dielectric measurement cells mentioned in previous work[4] and more recently in a cell for biochemical measurements.[5] Also reported are cells with temperature control on thin films[6] and for flow measuring.[7–9] However, in all these cases the cells are totally metallic and therefore nontransparent, making the study of the optical properties of whatever material is inside impossible.

In search of a suitable solution, some time ago we developed[4] a system consisting of a large (0.2 m^3) chamber of transparent acrylic sheets, wherein warm air was blown on a Chatelain prism that served as a measuring cell.[10,11]. The temperature in the cell was obtained by measuring the thermistor resistance on a Wheatstone bridge. A second thermistor was placed close to the cell and connected to a specially designed electronic circuit that controlled the heat of the air blown into the chamber.

Although useful and simpler to handle, this system still required measuring thermistor resistances and their conversion to temperatures. Initially a Wheatstone bridge was used, but it was later replaced by a Multimeter with an RS-232 connection that allowed us to follow the process through a PC.

However, even with these improvements the system still had several important disadvantages. First, a rather large chamber was needed to house the whole system, with hot air being circulated through it. This required a double temperature control, inside the cell and outside close to it. Second, the diameter of the thermistor (though quite small, 0.02 mm outer diameter) required a rather large angle in the prism ($7°-8°$), so that only a small part of the sample could be in contact with it. Third, the conversion of thermistor resistance to temperatures was cumbersome.

Our system is based on a copper cell consisting of two vertical plates, 3 mm thick, both with a 12×12 mm square opening, that are placed on a $4\times4\times0.5$ cm thick acrylic slab. One of the plates is fixed, while the other can be moved horizontally to adjust the intervening space. The Chatelain prism, placed inside, is then held firmly to make good con-

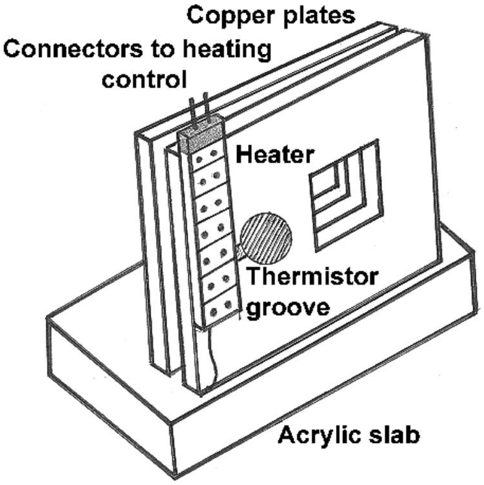

FIG. 1. The complete copper cell.

tact with the copper plates. This is important to ensure adequate heat transfer. The movable plate has a groove to hold a thin thermistor[12] (see Figs. 1 and 2) and both have a (1 cm^2) square window for adequate optical observations.

The Chatelain prism consists of two thin ($30\times40\times1.0$ mm) microscope glass slides that are kept separated at one end by a $4\times20\times2$ mm Teflon separator. Since the plates are of standard size and thickness, the angle is very easy to calculate by measuring with a caliper. In this manner quite a small angle (2°) is obtained.

The cell is heated with a miniaturized device that creates a hot air current inside a small acrylic chamber ($2\times5\times20$ cm) (Fig. 3), and its temperature is read on the thermistor connected thermally on the fixed copper plate. The heating system creates a temperature ramp that goes from room temperature (~288.15 K) to about 313.15 K over a period of 3 h. An approximate rate of 0.14° per minute is obtained. This scan rate was chosen because it was found to be adequate, but it can be either increased or reduced through changes in the firmware. Since the purpose of this work is to scan optical properties over the whole range of a mesophase, no provision is made to keep the temperature constant.

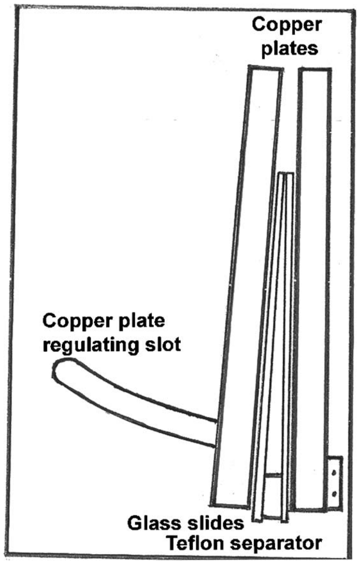

FIG. 2. View of the copper plates, glass slides, and Teflon separator.

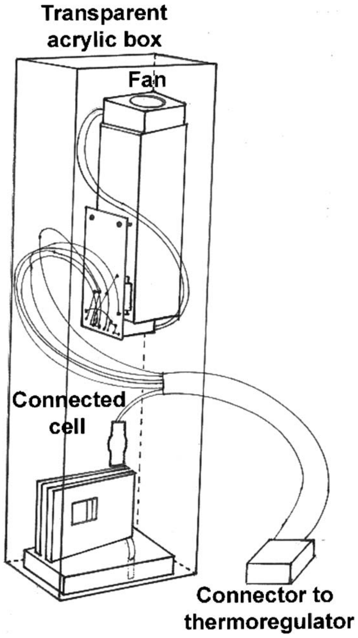

FIG. 3. Heating box with the cell.

The block diagram of the heating circuit is shown in Fig. 4, where it can be seen that the thermistor[12] is connected in series with R to a 2.5 V stabilized source. The divider voltage is digitized through a 12 bit A/D converter, adequate for a 0.01° resolution, and the temperature is read between the indicated values (288.15–233.15 K). The firmware does the necessary calculations both to linearize the thermistor and to provide four-figure temperature readings (two whole and two decimals) in seven-segment, four-digit light-emitting diodes (LEDs). In this way no additional calculations are necessary because the cell temperature is read directly on the display. These calculations are carried out as follows: The relationship between the temperature and the thermistor resistance is $R_t=A^*\exp(B/T)$.[1] Here, A and B are the individual thermistor constants that are derived from precise measurements in the 20-40 °C interval. Then, for any two temperatures (T_1 and T_2), the equation becomes $R_t1=A^*\exp(B/T_1)$ and $R_t2=A^*\exp(B/T_2)$, so that $B=\ln(R_t1/R_t2)[(1/T_1)-(1/T_2)]$ and $A=R_t1^*\exp(-B/T_1)$, T being the absolute temperature.

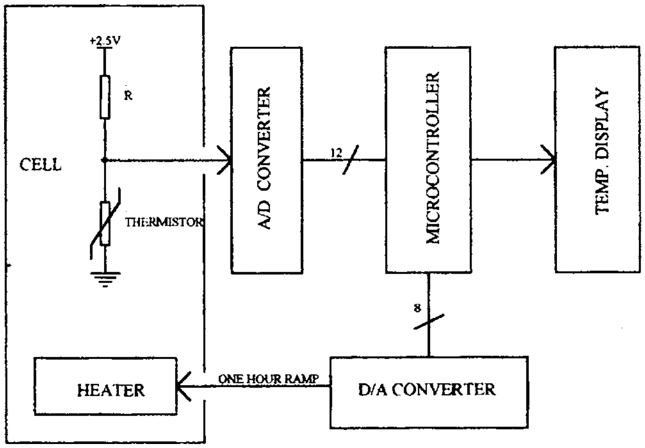

FIG. 4. Block diagram of the temperature control.

Therefore, from Eq. (1) the temperature T as a function of resistance R becomes $T=B/[\ln(R_t/T)]$.[2]

The resistance values R are determined connecting the thermistor, in series with a metal film resistance, to a 2.5 V stabilized source. Then,

$$V_t = 2.5^{*}R_t/(R_s + R_t) \tag{1}$$

then

$$R_t = V^{*}R_s/(2.5 - V_t). \tag{2}$$

V is measured with a 12 bit A/D convertor and this value is replaced in Eq. (2), leading to the desired temperature with a 0.01 K resolution. The needed calculations are contained in the microcontroller software. The precision is of the order of ±1 °C over the whole range, but it can be calibrated to ±0.1 °C in an interval below ±3 °C. The thermistors have a mean resistance of 100 kOhms at 20 °C.

The program was written in C language, using the function incorporated in the library to calculate natural logarithms. The executable has an approximate length of 2 KB.

The heater itself is an ordinary power transistor (Q1), controlled by an operational (U4) so that the current in Q1 is proportional to the tension on pin3 of U4. The 3 h ramp is generated digitally through firmware in the microcontroller U1 that is converted to analog through U3 for control. This transistor is mounted on an aluminum square pipe, of 2.45 cm section and 6 cm long that carries a small CPU fan on one end. The device blows hot air on the cell. The cell and heater are enclosed within an acrylic box high enough that the fan is not affected by a magnetic field acting on the cell.

The system is calibrated once measuring the temperature inside the cell at the spot where the sample will be, using another thermistor. The difference found with that indicated in the LEDs of the electronic device is then used to correct the systematic error in the thermistor on the copper plate. This provides adequate calibration so that the reading on the display can be adjusted to show the actual temperature inside the cell. The calibration is reliable as long as the thermistor remains whole. Due to their fragility, these thermistors may break, in which case a new calibration is required.

The operation is very simple because, once the cell is prepared with the sample to be examined, it is allowed to attain room temperature. This can be established by connecting the thermistor to the circuit without placing the acrylic box on the cell. In this way warm air is not blown on it and the temperature on the thermistor can be read. The cell is then covered with the box and the ramp is started, so that warm air is blown on it and the rise in temperature can be read directly on the circuit display. So, the large chamber used previously has now been successfully discarded.

With this procedure and device, the temperature inside the cell, although read directly on the display to 0.01 K, will remain stable for periods of 5 min, which provides enough time to make reliable angle readings on the goniometer. This is a simpler and more effective way than that of the instrument used previously because it requires only one thermistor with its electronic circuit and, as indicated above, a second thermistor is used only once for calibration.

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