RAPID DETECTION AND IDENTIFICATION OF LIQUID CRYSTAL STATES BY LIGHT SCATTERING

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Thermotropic mesophases can be detected by the light scattering properties of these phases. This article deals with a rapid scan technique for the detection and characterization of these mesophases by measuring light scattering as a function of temperature. From the number of compounds studied it is apparent that the method allows for a rapid and reasonably precise determination of transition temperatures. In addition, the method is found to be useful in the characterization of mesophases, in particular the nematic phase.

Historically, the discovery of mesophases is intimately associated with the light scattering properties of mesophases [1–5]. Early investigators observed that melting, as determined by the fluid properties of the substance, often yielded a definitely opaque or turbid fluid state. On increasing the temperature the opacity or turbidity abruptly disappears yielding a true isotropic liquid. These turbid or opaque states were shown later to be true mesophases. By careful observation of changes in turbidity as a function of temperature one can determine mesomorphic transition temperatures with a fair degree of accuracy. However, there are several limitations connected with the visual determination of mesomorphic transition temperatures amongst which is the considerable eye strain involved in such determinations.

With the advent of modern electronic temperature programmers, the automatic determination of transition temperatures has now become feasible. The principle of these methods involves the recording as a function of temperature of any physical property which undergoes an abrupt change at the transition temperature. Examples of this are the techniques of differential thermal analysis and differential scanning calorimetry where one essentially measures the enthalpy changes occurring at each transition.

In this paper, we will describe a new automated rapid scanning technique involving the measurement of light scattering.

Description of technique

The apparatus that we have used utilizes the Mettler Model FP2 melting point apparatus with linear cooling mode capability, but any apparatus of similar design can be used. Essentially, the apparatus consists of a linear temperature
programmer and a furnace with a built-in tungsten light source and a solid state photodetector (see Fig. 1). An output from the photodetector is available for direct connection to the y axis of any x – y 10 mV recorder. The sample is contained in capillaries, available from the Mettler Co., which are 1 mm in diameter and 9 cm in length. Again, any similar thin walled capillary can be used. A thin chromel–alumel thermocouple is inserted in the sample tube along with the sample and the entire assembly then placed in the furnace between the light source and photodetector. The thermocouple signal is then amplified and connected to the x-axis of the x – y recorder. We, therefore, have the capability of monitoring the intensity of light transmitted by the sample as a function of the temperature of the sample. It should be pointed out that the change in intensity of transmitted light is a function of the light scattering properties of the sample and not its light absorbing properties. The apparatus has the capability of monitoring light transmission in both the heating and cooling mode.

Sample handling is quite important in this technique and a detailed description of this procedure follows. The capillary is filled to a depth of 6 mm by the usual techniques, and then the fine chromel–alumel thermocouple inserted such that the tip of thermocouple is 4 mm from the bottom of the capillary. The capillary tube is then warmed, by a flow of hot air, until the sample is just molten and fills the lower portion of the tube. At this point, the sample tube is cooled slowly to room temperature and allowed to stand for at least one hour, so as to minimize any supercooling effects. It is absolutely essential during this warming and cooling process that air bubbles be not trapped in the sample. We have found that gentle tapping of the tube while the sample is in the molten state easily removes any trapped gases. The final capillary-thermocouple assembly will have an appearance.

Fig. 1. Block diagram of light scattering apparatus