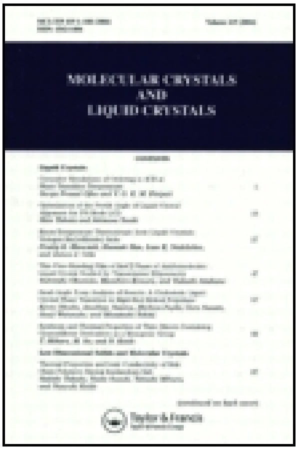


Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl20

Accurate Method for the Determination of Phase Transitions in Liquid Crystals with Photoacoustic Technique

S. Lakshminarayana [a], K. Gouthami [b], V. G. K. M. Pisipati [c], N. V. S. Rao [d] & G. Venkata Rao [e]

[a] Department of Electronics & Communications Engineering, Koneru Lakshmaiah University College of Engineering, Vaddeswaram, India

[b] Department of Electronics and Communications Engineering, Nalanda Institue of Engineering and Technology, Kantepudi, India

[c] S.S.D. Polymers, APIE, Machilipatnam, India

[d] Department of Chemistry, Silchar University, Assam, India

[e] PATNI Computer Systems Ltd, Thane, India

Version of record first published: 05 Oct 2009

To cite this article: S. Lakshminarayana, K. Gouthami, V. G. K. M. Pisipati, N. V. S. Rao & G. Venkata Rao (2009): Accurate Method for the Determination of Phase Transitions in Liquid Crystals with Photoacoustic Technique, Molecular Crystals and Liquid Crystals, 511:1, 50/[1520]-58/[1528]

To link to this article: http://dx.doi.org/10.1080/15421400903048578

Mol. Cryst. Liq. Cryst., Vol. 511, pp. 50/[1520]–58/[1528], 2009

ISSN: 1542-1406 print/1563-5287 online
DOI: 10.1080/15421400903048578

Accurate Method for the Determination of Phase Transitions in Liquid Crystals with Photoacoustic Technique

S. Lakshminarayana[1], K. Gouthami[2], V. G. K. M. Pisipati[3], N. V. S. Rao[4], and G. Venkata Rao[5]
[1]Department of Electronics & Communications Engineering, Koneru Lakshmaiah University College of Engineering, Vaddeswaram, India
[2]Department of Electronics and Communications Engineering, Nalanda Institue of Engineering and Technology, Kantepudi, India
[3]S.S.D. Polymers, APIE, Machilipatnam, India
[4]Department of Chemistry, Silchar University, Assam, India
[5]PATNI Computer Systems Ltd., Thane, India

A compact Photoacoustic spectrometer (PAS) was fabricated, for the determination of phase transitions in liquid crystals (LCs). Photoacoustic (PA) technique is a powerful tool for the determination of phase transitions in solids, liquids and gasses compared to other techniques like Raman spectroscopy, Newton scattering, NMR & ultrasound studies and DSC. PA signal information is used to investigate the variations in the optical and thermal properties of the materials during phase transitions. The advantage of PA technique is its simplicity in the sample preparation, and its accuracy in the phase transition studies. The instrumentation details and results of phase transition studies on p-pentyloxy benzoic acid (5OBA), N(4-n-hexadcyloxysalicylidene)-2-methylbutyl-4′-amino-cinnamate, 16O(OH)2MBC and N-(4-n-butyloxysalicylidene)-4′-n-Ketylaniline, 4O(OH)5 are presented. The advantages of PA technique are elaborated.

Keywords: liquid crystal; phase transitions; photoacoustic; resonance cell

1. INTRODUCTION

In the field of liquid crystal research, it is common to look at the new compounds through the design and synthesis [1–4]. The thermal measurements (polarizing microscope attached with hot stage and differential scanning calorimeter) play an important role in finding and

Address correspondence to S. Lakshminarayana, Department of Electronics & Communications Engineering, Koneru Lakshmaiah University College of Engineering, Vaddeswaram-522502, Guntur District, India. E-mail: slakshminarayana@hotmail.com

characterizing the different phases and phase transitions in the liquid crystal compounds [5]. Photoacoustic effect, mirage effect and photon transmission methods are some of the optical methods used for the study of phase transitions in liquid crystals [6–10]. The optical techniques, being highly sensitive, less time consuming and applicable over a wide spectral region, have produced interesting results in liquid crystal characterization [11]. This manuscript reports the experimental results of the detection of phase transitions in three liquid crystals using PA technique. Photoacoustic spectroscopy is now commonly used in the analysis of a variety of materials as it is a non-destructive technique. Further, it offers minimal or no sample preparation, and ability to look at opaque and scattering samples also. It has the capability to perform depth profiling experiments. PAS can be used for both qualitative and quantitative analysis. In particular, depth profiling experiments are also useful for the characterization of surface-coated and laminar materials and for studies of weathering, aging, curing, and the diffusion of species into or out of a polymer matrix. It is especially useful for the study of electro-optic effects in different layers of liquid crystals.

2. PRINCIPLE

The origin of Photoacoustic spectroscopy (PAS) dates back to the discovery of the Photoacoustic effect by Alexander Graham Bell in 1880. Bell found that when light was focused onto thin diaphragms, and interrupted periodically, sound was emitted. In latter experiments, Bell studied the sounds produced by the irradiation of various solid samples in a brass cavity sealed with a glass window.

Practical use of the photoacoustic effect for condensed phase materials had to wait for advances in instrumentation and theory. In 1973, PAS was rediscovered simultaneously by A. Rosencwaig [12] at Bell Laboratories and by A. G. Parker at Johns Hopkins University. A general theory for the Photoacoustic effect was developed by Rosencwaig and Gersho and is commonly referred to as the RG Model.

3. DESCRIPTION

Figure 1 shows the Block Diagram of PA Spectrometer. Figure 2 shows the cross-sectional view of the resonance PA Cell. The sample is placed in a small aluminum pan and kept inside the resonance cavity of the PA cell. The temperature was controlled by a commercial temperature controller and a heating cartridge was inserted through a hole in the PA resonance cell. The temperature was measured with a digital

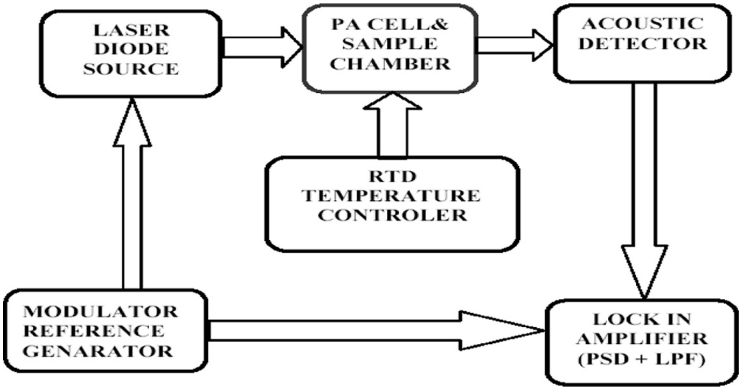


FIGURE 1 Block diagram of photo acoustic spectrometer.

thermometer, at intervals of 0.5°C, whose probe is inserted through another hole of the PA resonance cell. The PA signal picked up by a low noise microphone is amplified by a low noise preamplifier LA3161 and connected to the input channel of the lock-in amplifier. The reference signal to the lock-in amplifier is the modulating frequency of the laser diode. The laser diodes used are 660 nm, 50 mW, 808 nm 500 mW, driven by a Laser diode driver iC-WJ, (iC Haus, Germany). The laser diode driver circuit details are given in Figure 3.

A modulation frequency of 30 Hz was used for the optimum performance of the PA cell. The performance of the instrument was checked by placing a glass plate coated with carbon soot in the cavity and

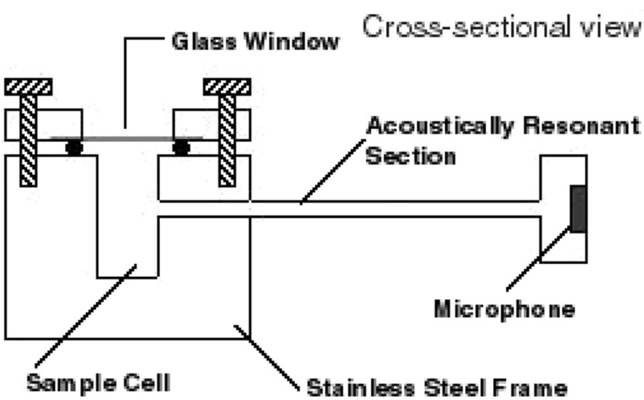


FIGURE 2 Resonance photoacoustic cell.

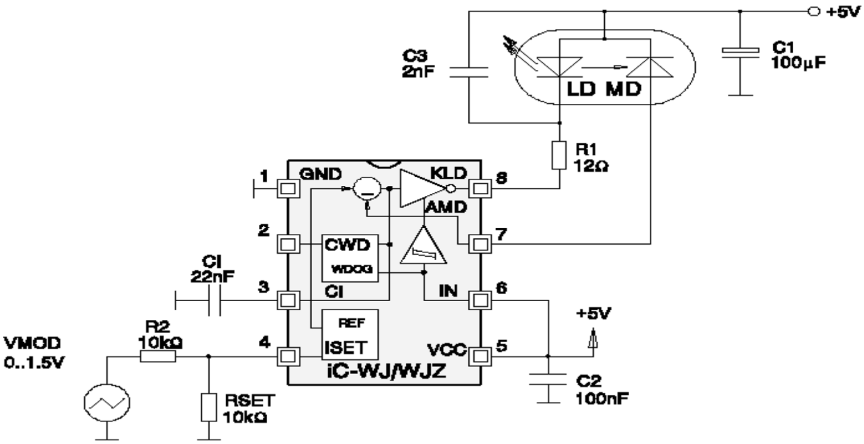


FIGURE 3 Laser diode driver.

observing the PA signal. Vallabhan *et al.* [13], developed open Photo acoustic cell for the thermal characterization of liquid crystals.

4. EXPERIMENT AND OBSERVATIONS

Photoacoustic Studies of Phase Transitions in Liquid Crystals

1. *p-Pentyloxy Benzoic Acid (5OBA)*

The PA spectra of *p*-pentyloxy benzoic acid (5OBA) is shown in Figure 4. The compound exhibits a monovariant Nematic phase. The compound is obtained from M/S Frinton laboratories [14], Vineland, New Jersey, U.S.A. Pure 5OBA [15] compound shows threaded marble

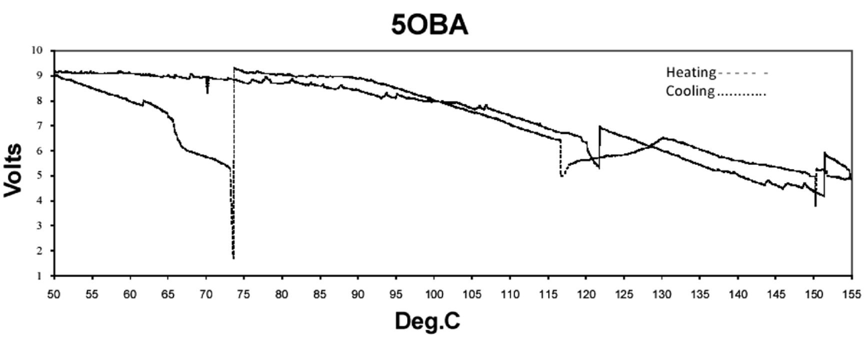
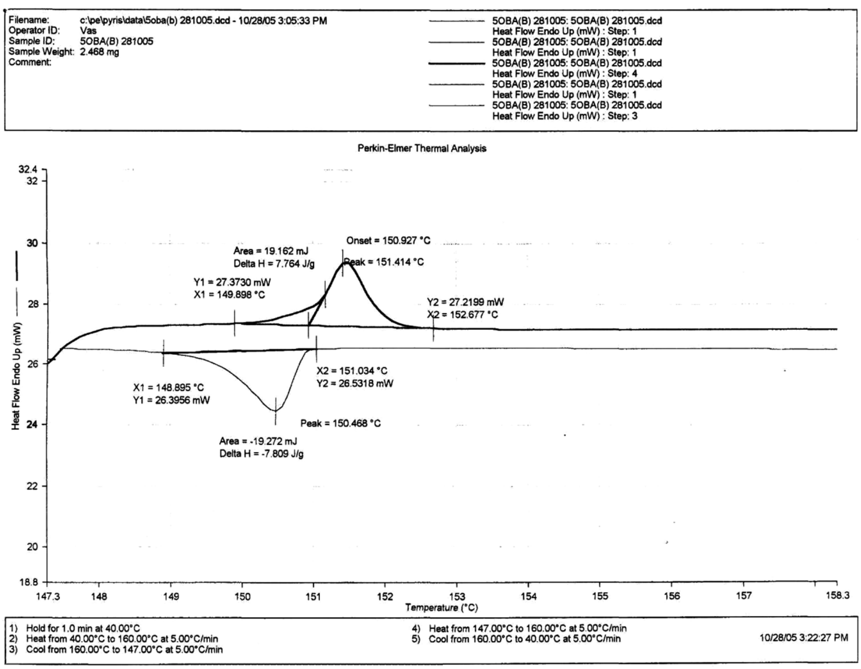


FIGURE 4 Photoacoustic signal of 5OBA.

TABLE 1 Phase Transitions of 5OBA (Present Work)

PAS	Solid phase	Solid to nematic	Nematic to isotropic
Heating	70.1°	121.7°	151.4°
Cooling	73.5°	116.7°	150.2°
DSC			
Heating	—	121.2°	151.4°
Cooling	—	113.6°	150.4°
Thermal Microscopy			
Heating	—	122.8°	148°
Cooling	—	121.6°	166.5°

Nematic phase between (122.8°C–148°C). During cooling, the nematic droplets separated from the isotropic phase at 146.5°C, and slowly they combine to form Nematic phase and this phase remains up to 121.6°C. On further cooling it becomes as solid below 121.60°C. The transitions observed through PAS agree well with those obtained from



Scanning rate: 10^0C/min.

FIGURE 5 DSC data of 5OBA.

TABLE 2 Consolidated Data of 4O(OH) 5

Structure	PAS		DSC		TM	
Crystal	Heating Below Room Temperature	Cooling	Heating Below Room Temperature	Cooling	Heating Below Room Temperature	Cooling
	42.7°		42.871		42.87°	
Smectic-C	51.3°	50.8°	51.461	50.458	51.46°	50.45°
Smectic-A	74.1°	72°	74.227	71.949	74.23°	71.95°
Nematic	93.8°	94.7°	94.718	94.016	94.71°	94.1°
Isotropic					At Above 94.71°	Above 95°

TABLE 3 Consolidated Data of 16O(OH)2MBC (Not Published)

Structure	PAS		DSC		TM	
	Heating	Cooling	Heating	Cooling	Heating	Cooling
Crystal	67.1°	54.2°	67.004°	53.279	67°	53.27°
Smectic-C*	99.2°	98°	99°		Absent	Absent
Smectic- A	137.6°	136.5°	137.501°	136.422	137.5°	136. 4°
Isotropic	At Above 137.6°		At Above 137°		At Above 137.5°	Above 137°

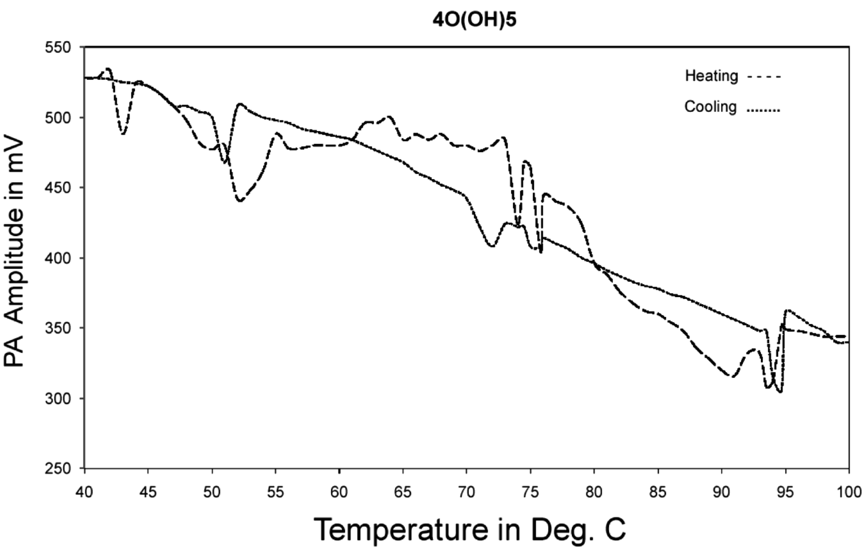


FIGURE 6 PA spectra of 4(OH)5.

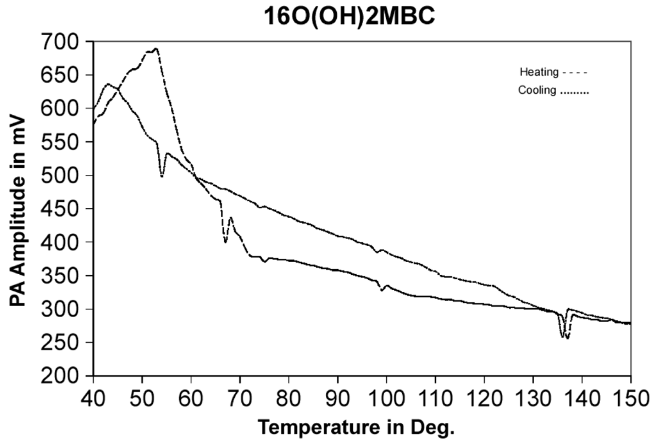


FIGURE 7 PA Spectra of 16O(OH)2MBC.

the DSC, and polarizing microscope and the results are shown in the Table 1. The isotropic to Nematic transition observed through DSC is given in Figure 5.

To further test the effectiveness of PA method, the experiment was repeated with other wavelength 808 nm, and without Lock-In amplifier using two other LC samples viz., 4O(OH)5 and 16O(OH)2MBC [16].

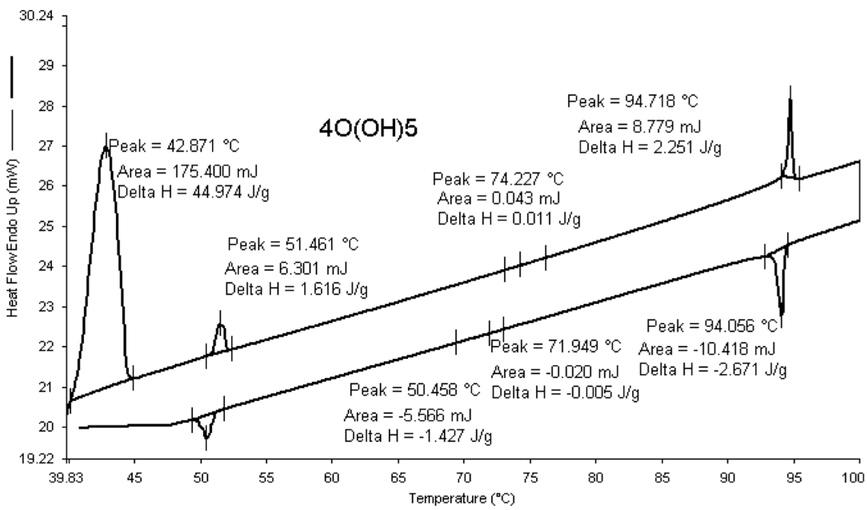


FIGURE 8 DSC data of 4O(OH)5.

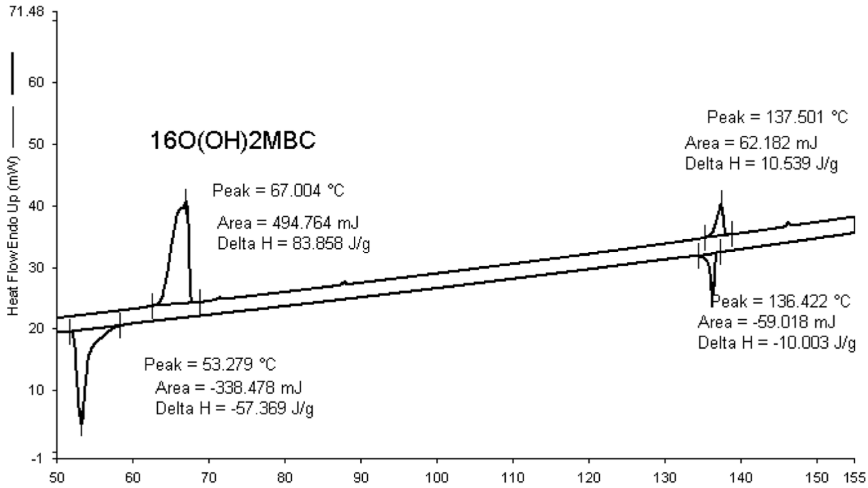


FIGURE 9 DSC data of 16O(OH)2MBC.

The characteristic PA response of the 4O(OH)5 compound during heating and cooling cycles in the temperature range of 30°C to 100°C are studied and the results obtained by various techniques are shown in Table 2. The phase transitions in 4O(OH)5 compound in heating and cooling is shown in Figure 6.

The characteristic PA response of the 16O(OH)2MBC compound during heating and cooling cycles in the temperature range of 30°C to 150°C are studied and the results obtained by various techniques are shown in Table 3. The phase transitions in 16O(OH)2MBC compound in heating and cooling is shown in Figure 7.

The DSC data of compounds, 4O(OH)5, and 16O(OH)2MBC, was shown in Figures 8 and 9.

CONCLUSIONS

1. The normal behavior of the PA cell is studied by the amplitude response of the PA cell with carbon soot. The PA signal obtained from this cell is strong, and is detected over a wide thermal range from 30°C to 150°C.
2. The ability of the instrument to detect the phase transition of *p*-pentyloxy benzoic acid (5OBA) at 121.76°C, 151.4°C and 150.4°C, 113.6°C, simultaneously both during heating and cooling cycles indicates the suitability of this setup for PA studies of solids,

liquids and liquid crystals, over a wide temperature range and with a high degree of accuracy.

3. The PA spectra of 4O(OH)5, and 16O(OH)2MBC, shows the reliability of PA technique, compared to other methods.

REFERENCES

[1] Ajeetha, N. & Pisipati, V. G. K. M. (2003). *Z. Naturforsch*, *58a*, 735.
[2] Ajeetha, N. & Pisipati, V. G. K. M. (2005). *Z. Naturforsch*, *60a*, 207.
[3] Ajeetha, N., Potukuchi, D. M., & Pisipati, V. G. K. M. (2005). *Phase Transitions*, *78*, 369.
[4] Prabhu, C. R., Lakshiminarayana, S., & Pisipati, V. G. K. M. (2004). *Z. Naturforsch*, *59a*, 537.
[5] Padmaja, S., Srinivasulu, M., & Pisipati, V. G. K. M. (2003). *Z. Naturforsch*, *58a*, 573.
[6] George, N. A., Vallabhan, C. P. G., Nampoori, V. P. N., George, A. K., & Radhakrishnan, P. (2000). *J. Phys. D: Appl. Phys.*, *33*, 3228.
[7] Rajasree, K., Vidyalal, V., Radhakrishnan, P., Nampoori, V. P. N., Vallabhan, C. P. G., & George, A. K. (1998). *Mater. Lett.*, *36*, 76.
[8] Nagaraja, C. Design and development of PC based Photoacoustic spectrometer for investigation of phase transitions in Liquid Crystals, Ph.D. thesis submitted to S. K. University, 2000.
[9] Ozbek, H., Yildiz, S., & Pekcan, O. (1999). *Phys. Rev.*, *E59*, 6798.
[10] Puccetti, G. & Leblanc, R. M. (1998). *J. Chem. Phys.*, *108*, 7258.
[11] Sahraoui, A. H., Kolinsky, C., Delenclos, S., Daoudi, A., & Buisine, J. M. (1997). *J. Appl. Phys.*, *82*, 6209.
[12] Rosencwaig, A. (1980). *Photo Acoustics and Photo Acoustic Spectroscopy*, John Wiley & Sons: New York.
[13] Vallabhan, C. P. G., *et al.* (2001). *Appl. Phys. B*, *73*, 145–149.
[14] Frinton laboratories, Vinland, Newjersy USA. Gray, G. W. (1953). *Nature*, *172*, 1137.
[15] Rajeswari, A. V. M. Phil thesis Acharya Nagarjuna University, India.
[16] Prof. N. V. S. Rao. Not published. Department of Chemistry, Silchar University, Assam, India.