CHAPTER 6: RESULTS AND DISCUSSIONS (RESONATING TYPE PHOTOACOUSTIC SPECTROMETER)

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6.1 Introduction

The performance of microcontroller based photoacoustic spectrometer system, designed and fabricated for the present work is tested with carbon black sample. Both the modulation frequency and temperature response of the PA cell are studied. For standardization of the photoacoustic system a diode laser of 50mW power and a wavelength of 808nm is used as radiation source. The laser is chopped by TTL reference signal generated by lock-in amplifier. The photoacoustic signal is detected by an electret microphone together with pre-amplifier, filter and lock-in amplifier indigenously designed and fabricated for the present work. The calibration of the PA cell is carried out with a carbon black (prepared from the soot) as the reference sample. The carbon soot is coated on glass plate of 1.0cm diameter and thickness of 1mm is used.

6.2 Standardization of the PA Cell with Carbon Black

The standardization of the PA cell is done by measuring the photoacoustic signal amplitude of the standard sample i.e. carbon black, as a function of temperature. The sample characteristics are studied as a function of temperature by employing a microcontroller based PID temperature controller. Fig 6.1 shows the dependence of photoacoustic signal amplitude as a function of temperature. The signal behavior is studied from room temperature 27°C to 200°C. It is observed that the amplitude of the photoacoustic signal decreases continuously with increase of temperature as can be seen from the figure. This behavior is qualitatively in good agreement with the results reported by Vallabhan and Betchold [1-2].

6.3 Frequency Response of PA cell with Carbon Black

Before attempting to the application of the instrument, it is essential to determine the volume resonance of the PA cell. For this purpose, the carbon soot is coated on the glass plate of 1.0cm diameter, which acts as a sample, is placed in the sample cavity of the PA cell. The frequency response of the cell is studied at room temperature (of 27°C). Fig 6.2 shows the frequency response of the PA cell. From this response it is found to exhibit a strong resonance peak around 350Hz. The modulating frequency is varied from 10 Hz to 600 Hz in steps of 10 Hz and amplitude of the signal is measured as function of frequency. Graph is drawn between the frequency and amplitude of the signal. The graph
shows that the signal amplitude is the maximum at 352 Hz. This sharp increase in the amplitude of the signal around 352 Hz indicates the characteristic volume resonance of the PA cell. The frequency response characteristic observed for the cell exhibits similar characteristic as reported by Vallabhan and Betchold [1-2]. Hence, a modulation frequency of 352 Hz which gives a good PA signal has been chosen. The above results obtained through the studies on the standard sample, establish the reliability of the PA cell and the microcontroller based photoacoustic spectrometer for studying the phase transitions of the samples. Hence, the system is applied to study the phase transitions of various compounds. The results of those are discussed in the foregoing sections.

![Graph showing Amplitude variation of the PA signal with temperature (Carbon)](image_url)

**Fig. 6.1** Amplitude variation of the PA signal with temperature (Carbon)
6.4 Application of PAS for Phase Transition Studies

In the present investigation the microcontroller based photoacoustic spectrometer designed and fabricated is employed for identification of phase transitions of some organic/inorganic compounds such as acetaldehyde, benzoic acid, β-napthol, cinnamic acid, glucose, naphthalene, oxalic acid, urea, and sulphor.

a. Acetaldihyde

The amplitude variations with temperature for the compound acetaldehyde observed through PAS technique is presented in Fig. 6.3(a) for heating cycle. It is observed that there are sudden changes in the PA signal amplitude at temperature 121.0 °C which is solid-liquid transition temperature of acetaldihyde. The DSC graph for the same compound is presented in Fig. 6.3(b) for heating cycle, here the sudden change in the PA signal amplitude is observed at temperature 119.670 °C. The results of the PAS and DSC studies on acetaldihyde are consolidated in Table 6.1
Fig. 6.3 (a)

Fig. 6.3 (b)
b. Benzoic Acid

The amplitude variations with temperature for the compound benzoic acid observed through PAS technique is presented in Fig. 6.4 (a) for heating cycle. It is observed that there are sudden changes in the PA signal amplitude at two temperatures 125.3 °C and 150.5 °C. The DSC graph for the same compound is presented in Fig. 6.4(b) for heating cycle. From the graph we can observe that the sudden change in the PA signal amplitude is at only one particular temperature 124.3 °C. From the two figures 6.4 (a) and (b) we can observe that using PAS technique we have obtained two transitions at 125.3 °C and 150.5 °C where as in DSC only one transition at 124.30 °C is detected. The results of the PAS and DSC studies on benzoic acid are consolidated in Table 6.1

![Graph of Amplitude vs Temperature for Benzoic Acid](image)

Fig. 6.4 (a)
c. β-Napthol

The amplitude variations with temperature for the compound β-napthol observed through PAS technique is presented in Fig. 6.5(a) for heating cycle. It is observed that there are sudden changes in the PA signal amplitude at four different temperatures 125.9°C, 150.1°C, 175.28°C, and 190.21°C. The DSC graph for the same compound is presented in Fig. 6.5(b) for heating cycle. From the graph we can observe that the sudden change in the PA signal amplitude is observed at only one particular temperature 126.45°C. From the two figures 6.5 (a) and (b) we can observe that using PAS technique we have obtained four transitions where as in DSC only one transition is detected. The results of the PAS and DSC studies on β-napthol are consolidated in Table 6.1
**β-Napthol**

Fig. 6.5 (a)

Fig. 6.5 (b)
d. Cinnamic Acid

The amplitude variations with temperature for the compound cinnamic acid observed through PAS technique is presented in Fig. 6.6(a) for heating cycle. It is observed that there are sudden changes in the PA signal amplitude at three different temperatures 131.9°C, 168.1°C, and 197.16°C. The DSC graph for the same compound is presented in Fig. 6.6(b) for heating cycle. From the graph we can observe that the sudden change in the PA signal amplitude is at only one particular temperature 133.65°C. From the two figure 6.6 (a) and (b) we can observe that using PAS technique we have obtained three transitions at 131.9°C, 168.1°C, and 197.16°C. where as in DSC only one transition at 133.65°C is detected. The results of the PAS and DSC studies on cinnamic acid are consolidated in Table 6.1

![Cinnamic acid graph](image)
The amplitude variations with temperature for the compound Glucose observed through PAS technique is presented in Fig. 6.7 (a) for heating cycle. It is observed that there are sudden changes in the PA signal amplitude at two temperatures 79.18 °C and 149.13 °C. The DSC graph for the same compound is presented in Fig. 6.7(b) for heating cycle. From the graph we can observe that the sudden changes in the PA signal amplitude are at two temperature 77.38°C and 152.48°C. From the two figures 6.7(a) and (b) we can observe that using DSC, two transitions are obtained, and these two transitions are detected by PAS technique. The results of the PAS and DSC studies on Glucose are consolidated in Table 6.1
f. Naphthalene

The amplitude variations with temperature for the compound naphthalene observed through PAS technique is presented in Fig. 6.8(a) for heating cycle. It is observed that there are sudden changes in the PA signal amplitude at temperature 83.5°C which is solid-liquid transition temperature of naphthalene. The DSC graph for the same compound naphthalene is presented in Fig. 6.8(b) for heating cycle. From the graph 6.8(b) we can
observe that the sudden change in the PA signal amplitude is at one particular temperature 85.5°C. From the two graphs 6.8(a) and (b) we observe that the transition temperature detected by using PAS technique is on par with the DSC detected transition temperature. The results of the PAS and DSC studies on naphthalene are consolidated in Table 6.1

![Graph](image_url)

**Fig. 6.8 (a)**

![Graph](image_url)

**Fig. 6.8 (b)**
g. Oxalic Acid

The amplitude variations with temperature for the compound oxalic acid observed through PAS technique is presented in Fig. 6.9(a) for heating cycle. It is observed that there are sudden changes in the PA signal amplitude at four different temperatures 102.89°C, 115.75°C, 121.97 °C, and 140.38°C. The DSC graph for the same compound is presented in Fig. 6.9(b) for heating cycle. From the graph 6.9(b) we can observe that the sudden change in the PA signal amplitude is at four particular temperatures 102.8°C, 114.7°C, 123.7°C and 147.3°C. From the two figure 6.9 (a) and (b) we can observe that using DSC four transitions are obtained, and these four transitions are detected by PAS technique. The results of the PAS and DSC studies on oxalic acid are consolidated in Table 6.1

![Fig. 6.9 (a)](attachment:image.png)
h. Urea

The amplitude variations with temperature for the compound urea observed through PAS technique is presented in Fig. 6.10(a) for heating cycle. It is observed that there are sudden changes in the PA signal amplitude at four different temperatures 114.0°C, 141.0°C, 165.4°C, and 184.3°C. The DSC graph for the same compound is presented in Fig. 6.10(b) for heating cycle. From the graph we can observe that the sudden change in the PA signal amplitude is at only two particular temperatures 116.14°C and 140.29°C. From the two figures 6.10(a) and (b) we can observe that using PAS technique we have obtained four transitions where as in DSC only two transitions are detected. The results of the PAS and DSC studies on urea are consolidated in Table 6.1.
Fig. 6.10 (a)

Fig. 6.10 (b)
i. Sulphor

The amplitude variations with temperature for the compound sulphor observed through PAS technique is presented in Fig. 6.11(a) for heating cycle. It is observed that there are sudden changes in the PA signal amplitude at one particular temperature 81°C. The results of the PAS studies on sulphor are consolidated in Table 6.1

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**Fig. 6.11 (a)**
Table 6.1 Comparative study of phase transitions in PAS & DSC

<table>
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<tr>
<th>Samples/Compounds</th>
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<th>Phase variation at Temperature (in °C)</th>
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<th>90 to 121</th>
<th>122 to 200</th>
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<td>DSC</td>
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<td>-</td>
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<td>-</td>
<td>124.3</td>
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<td>B-Naphthol</td>
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<td>Cinnamic acid</td>
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<td>131.9, 168.1°C, 197.16°C.</td>
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6.5 Conclusion

In the present study photoacoustic spectrometer is designed using microcontroller. The use of microcontroller eliminates the possible human error in the measurement and analysis. A high degree of accuracy and repeatability and wide frequency range of the measurements is achieved. The system is versatile, inexpensive and fast in nature and is fully automatic and highly reliable. The system so developed by the author was tested and standardized by certain standard samples. The photoacoustic spectrometer designed and fabricated in the present work is applied to study the phase transitions of organic/inorganic compounds such as acetaldehyde, benzoic acid, β-napthol, cinnamic acid, glucose, naphthalene, oxalic acid, urea, and sulphor. The phase transitions are effectively detected with designed photoacoustic spectrometer. The system is also able to detect some of the phase transitions which are not detected by DSC. The system is compact and reliable for studying the phase transitions of various samples. The design contains novel solutions to the problems arising from the use of microcontroller as central processor. The Programming flexibility, re-configurability, and low-cost are the good reasons for using microcontroller. The system is facilitated with serial interface to PC to store the data for further processing/analysis. The application of proposed instrument for PA studies showed as good performance as any commercial instrument available in the market.

Also, in the present research work the thermal properties of some materials are also studied by using non-resonating type photoacoustic spectrometer and the obtained results are on par with the results from literature survey. C8051F060 microcontroller based photoacoustic spectrometer is designed and fabricated. This approach makes the instrument very compact, low-cost, low power consuming, and versatile.
6.6 Future Scope for the Present Work

Design of C8051F060 microcontroller based photoacoustic spectrometer makes the system very compact, reliable and versatile. There is lot of scope for the extension of present work with respect to both hardware and software. The following suggestions are proposed.

- It is proposed to apply the designed microcontroller based photoacoustic spectrometer to study the phase transitions of various liquid crystal samples to test whether it can detect second order phase transitions as well.

- The study can be extended to design, development, and analysis of photoacoustic spectrometer by DSPIC IC which contains both DSP and microcontroller features.

- It is also proposed to develop photoacoustic spectrometer with FPGA/ CPLD in conjunction with DSP devices or Application Specific Integrated Circuits (ASICs).

- It is proposed to develop user-friendly interface system by extending the work using LabVIEW or MATLAB software.

The results obtained through this technique are compared with the results obtained with DSC measurements. The above results obtained through the PAS studies on the standard samples establish the reliability of the designed photoacoustic spectrometer for studying the phase transition of the samples. Hence, the system can be applied for phase transition studies of any solid samples.
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List of Publications

Papers Published in Journals


Papers Accepted in Journals


Papers Communicated in Journal


Papers Presented in Conferences/Symposia/Seminars

2. Participated in KSTA Regional Conference on Science and Technology for Harnessing National Resources towards Sustainable Development, held on 4-5\textsuperscript{th} Jan 2014, at University of Agricultural sciences, Raichur, KA.

3. Sapna and P. Bhaskar, “An overview on photoacoustic spectrometers”, submitted to Two days seminar for research scholars of Gulbarga University, to be held on 9th and 10th Feb 2017.
High-Gain Low-Noise Pre-Amplifier and Narrow Band-Pass Filter for Photoacoustic Spectrometer

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ABSTRACT: In this paper an attempt is made to design and develop a high-gain low-noise pre-amplifier and a narrow band-pass filter for photoacoustic spectrometer (PAS). A two-stage pre-amplifier with the gain of 1000 and a second order state variable narrow band-pass filter are designed and fabricated. The pre-amplifier and narrow band-pass filter circuits are designed with a low-noise op-amp (LM308). This arrangement improves the sensitivity and signal-to-noise ratio of the photoacoustic (PA) signal. The frequency response of the PA cell is studied with carbon-black and liquid crystal samples. The resonant frequency of the PA cell was found at 345Hz. The performance of the system is tested in both the presence and absence of modulated laser beam applied on the sample, and it is found that the signal quality has been improved with the pre-amplifier and narrow band-pass filter designed for the present study.

KEYWORDS: Pre-amplifier, Narrow band-pass filter, Photoacoustic, Spectrometer, low-noise.

1. INTRODUCTION

Modern measurement systems are the fruits of science and technology. The precise measurement systems play an important role in science and technology. No doubt several investigators have designed various pre-amplifiers and narrow band-pass filters through variety of techniques. The signal-to-noise ratio is the most important parameter that needs to be considered, when low level measurements or high resolution measurements are attempted [1-2]. A pre-amplifier conditions the signal coming from a transducer/detector for further amplification. Its primary function is to extract the signal from the detector without disturbing the intrinsic signal-to-noise ratio [3]. High-gain pre-amplifiers are commercially available, and can also be built using discrete components like FETs/MOSFETs and low noise integrated circuits like LM308, OP07 etc.

It has to be noted that, even with the best available electronic circuitry, the amplifier noise exceeds the signal generated by the transducer itself [4] resulting in a detection sensitivity far below the theoretical limits determined by the displacement of the microphone diaphragm [5]. Several circuits for microphone and piezoelectric transducer pre-amplifiers have been described by several investigators [6-8]. The use of operational amplifiers with low temperature coefficient and large feedback substantially reduces the gain drift with temperature in comparison to pre-amplifiers with FETs/MOSFETs in the input stage. There are electret microphones with built in MOSFET amplifiers which makes it easy to couple them to moderately high input impedance pre-amplifiers. The output of the pre-amplifier is normally applied to the narrow band-pass filter to improve the signal quality.

Filters can be broadly classified into two types viz., passive filters and active filters. One of the serious constraints with the passive filters is the necessity to always use the specified source and termination impedances, besides compensating for the heavy attenuation suffered by the signal. Cascading of the filters is also not a straightforward solution in spite of designing the filters so that their source and termination impedances are equal. Additional isolation amplifiers are to be interposed between cascaded filter sections. This introduces severe distortion in the filter characteristics due to non-ideal matching between source-end and the input filter-end section and the output-end of the filter and the input.
Design and fabrication of microcontroller based temperature control system for photoacoustic spectrometer

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Abstract—The design and fabrication of C8051F350 microcontroller based temperature control system for the photoacoustic spectrometer (PAS) is presented in this paper. PID controller is implemented to control the temperature of the photoacoustic cell. The algorithm is developed in embedded C in KIEL µV4 integrated development environment. The linear ramp input is applied as reference to the controller to vary the temperature of the PA cell linearly. This type of controller is essential to study the characteristics of sample as function of temperature. From the experimental results it is observed that the temperature controller gives the best tracking response for the ramp input. As C8051F350 is basically designed for developing single chip instruments, only few components are used in the fabrication of proposed temperature control system.

Keywords—Microcontroller, temperature, control, spectrometer

I. INTRODUCTION

Temperature is one of the most widely measured and frequently controlled variables in the industry. This is because quite often the processing and manufacturing of the desired product is possible only if the temperature is accurately measured and maintained. Further, it forms an important governing parameter in thermodynamic, heat transfer and a number of chemical reactions/operations. In addition, it is a fundamental quantity in much the same way as mass, length and time. The need for temperature control arises in various fields such as medical, biological, industrial and many times in basic scientific research and R&D laboratories. Many physical and chemical reactions are sensitive to temperature and consequently, temperature control is important in several industrial processes.

Temperature control also finds application in cryostats that are used to perform experiments at very low temperatures in the field of spectroscopy, X-ray diffraction and optical microscopy.

Temperature control plays a key role in many industrial processes; in addition, precision and quality in control of temperature is desirable. Hence several investigators [1-6] have designed and fabricated different types of temperature controllers. But the attempt to use SOC type microcontroller to control temperature are rather scarce in spite of several advantages that are associated with them. Temperature control is a process in which the temperature of an object is measured and the passage of heat energy into or out of the object is adjusted to achieve a desired temperature.

II. METHODOLOGY

The block diagram of microcontroller based temperature controller is shown in Fig 1. The Pt-100 is used to sense the temperature of the steel body, the sensed output is suitably modified with the help of a signal conditioner. Signal conditioner produces an analog voltage output which is converted into digital data by on-chip A/D converter of the microcontroller. The microcontroller computes the error by subtracting the measured temperature from the desired temperature (set point). Then the PID equations are solved by the microcontroller. The output of control program is a digital value which is fed to the actuator through on-chip D/A converter, the output of DAC is an analog voltage which is given to zero crossing detector. The zero crossing detector produces phase angle firing pulses to control power applied to the heater. The amount of heat, added to or removed from the strip heater, connected to the steel body, is decided by the DAC output which in turn controlled by the PID controller to maintain it at the desired temperature. The complete schematic diagram is shown in Fig.2. It consists of the following elements:

i. Temperature sensor
ii. Constant current source
iii. Instrumentation amplifier
iv. C8051F350 microcontroller board
v. Zero crossing detector
vi. Comparator
vii. Opto-isolator (opto-diac)

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Design and Development of Temperature Control System for Photoacoustic Studies

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Abstract - In the photoacoustic (PA) spectrometer applications, temperature control system is essential to study the phase transition of the samples as a function of temperature. In this paper, an attempt is made to design & develop C8051F020 microcontroller based temperature control system for photoacoustic spectrometer. The algorithm is developed in embedded 'C' in KIEL µP4 IDE. The temperature of the PA cell is measured by using Pt-100 temperature sensor. The temperature of the PA cell is controlled by using PID algorithm. Phase angle firing control technique is employed to control the power applied to the heater. The phase angle is controlled by the on-chip timer of the microcontroller. The PID controller is tuned to the best values to obtain desired output. The present temperature control system increases the temperature of the PA cell at the rate of 0.1° C/Min. This heating rate is enough to detect the phase transitions of the samples.

Keywords: Temperature, PID, PAS, KIEL, C8051F020

I. INTRODUCTION

Temperature is one of the most widely measured and frequently controlled physical variables in the industry. This is because quite often the processing and manufacturing of the desired product is possible only if the temperature is accurately measured and maintained. Further, it forms an important parameter in thermodynamic heat transfer and a number of chemical reactions/operations. The need for temperature control arises in various fields such as medical, biological, industrial, frequently in basic scientific research and R&D laboratories. Many physical and chemical reactions are sensitive to temperature and consequently, the temperature control is important in several industrial processes. Temperature control also finds application in cryostats that are used to perform experiments at very low temperatures in the field of spectroscopy, X-ray diffractometry and optical microscopy. Temperature control plays a key role in many industrial processes; in addition, precision and quality in control of temperature is desirable. Hence, several investigators [1-8] have designed and fabricated different types of temperature controllers. But the attempts to use microcontroller with improved features like C8051F020 microcontroller to control temperature are rather scarce in spite of several advantages that are associated with the use of such microcontroller.

Principle: The block diagram of microcontroller based temperature controller is shown in Fig 1.0. The temperature of the photoacoustic cell is sensed by Pt-100 and the output of the sensor is suitably modified using a signal conditioner. The signal conditioner produces an analog voltage which is converted into digital data by on-chip A/D converter of the microcontroller. The microcontroller computes the error by subtracting the measured temperature from the desired temperature (set point). Then the PID equations are solved by the microcontroller.

Fig. 1 Block diagram of C8051F020 microcontroller based temperature control system

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ABSTRACT: The paper describes the design and development of microcontroller based photoacoustic spectrometer (PAS) for studying the thermal properties of solid samples. In this system, an electret microphone is employed to achieve high signal to noise ratio. The pre-amplifier circuit is constructed using the IC AD620 due to its attractive features that are ideally suited for high accuracy and precision applications. IC AD630 is employed to design a lock-in amplifier. C8051F020 microcontroller has employed to acquire and process the signal from the photoacoustic cell. The frequency response of the cell is studied with carbon-black as a sample. The system is very compact and easy to carry the experiments.

KEYWORDS: Photoacoustic, Spectrometer, PA Cell, C8051F020:

I. INTRODUCTION

Most optical spectroscopy are based on the detection and analysis of the photons that are either transmitted or reflected by the sample. In cases where the sample is highly transparent and thus the absorption is very weak, or when the sample is highly opaque, it is very difficult to determine the amount of absorption of the photons by the sample through the conventional techniques. For the transparent samples, the derivative absorption technique was used but was still found to be quite inadequate. For opaque samples, techniques like Raman scattering [1], diffuse reflection [2], attenuated total reflection [3] etc were utilized but they too had the limitations that the wavelength regions to which they could be applied and the number of samples that could use these techniques were very small, and moreover, the data from these techniques were difficult to analyze. For such samples, which are weakly absorbing, highly scattering or opaque, and where measurements using conventional optical spectroscopic techniques cannot be used effectively, a new optical technique was introduced to spectroscopic detection and was called the PHOTOACOUSTIC SPECTROSCOPY (PAS). It was discovered by Alexander Graham Bell in 1880 [4, 5]. Names such as the photo-thermal opto-thermal and opto-acoustic effect are still used to describe this phenomenon. The basic difference of this effect from conventional optical spectroscopy is that here, the amount of energy absorbed following irradiation of the sample by the optical radiation by the sample is directly measured. The PA effect is essentially the generation and detection of acoustic or other thermo-elastic effects resulting from the absorption of any kind of modulated/pulsed electromagnetic radiation. The PA technique has gained wide importance as a powerful for the study of phase transition in solids. The wealth information contained in the PA signal can be used to investigate the variations in the optical and thermal properties of materials during phase transitions. The PA technique basically non-contact and non-destructive in nature and offers several advantages such as relative easy in sample preparation, the range of samples that can be studied etc. In general several reports have been available on the use of PA technique for the investigation of phase transitions in solids [6-15]. Few authors reported the application of PA effect to study the phase transitions and thermal properties of liquids crystals [16]. Several researchers have improved the basic PAS technique by incorporating several modifications in the PA cell design, radiation sources, detectors, data acquisition and processing systems etc [17]. The