CHAPTER 7

INSTRUMENTATION

7.1 Construction of a Low Cost Photoacoustic Spectrometer for Characterization of Materials

Introduction

Optical spectroscopy has been a scientific tool for over a century and it has proven invaluable in studies on reasonably clear media such as solutions and crystals and on specularly reflective surfaces. There are, however several instances where conventional transmission spectroscopy is in adequate even for the case of clear transparent materials. Such situations arise when one is attempting to measure very weak absorption. In addition to weakly absorbing materials, there are a great many non-gaseous substances, both organic and inorganic, that are not readily amenable to the conventional transmission or reflection modes of optical spectroscopy. These are usually light scattering materials; such as powders, amorphous solids, gel, smears and suspensions. To fill this gap, photoacoustic spectroscopy has come into existence. Photoacoustics (PA) is well known in research but it is less known to physics students at undergraduate and postgraduate levels. In this article a simple and low-cost experimental design of the photo acoustic spectrometer is explained which can be easily designed in any developing laboratories.

The principle of photoacoustic is the generation of acoustic energy from modulated light energy. The resulting energy propagates away from the source as acoustic waves. That is, the photoacoustic effect is the generation of acoustic waves in a sample resulting from the absorption of photons. The sample may be solid, liquid, gas, powder, gel or thin films. Alexander Graham Bell [1] discovered the photoacoustic in the year 1880. The PA effect is represented pictorially in figure 7.1.

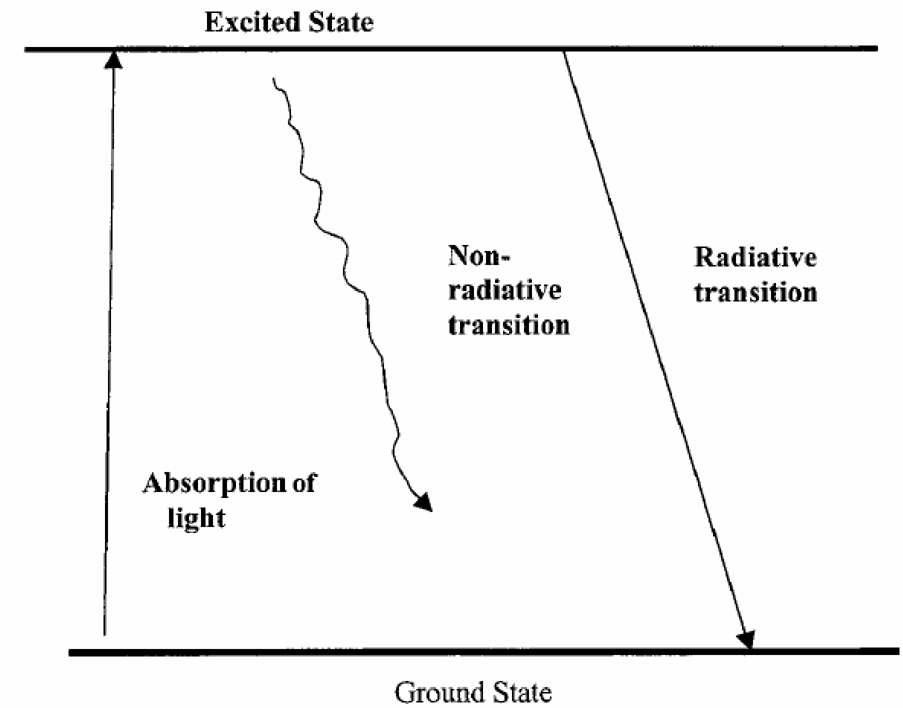
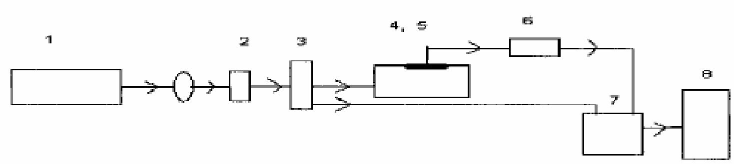


Figure 7.1 Principle of Photoacoustic Spectrum

Experimental

Construction of PA spectrometer All the parts of the PA spectrometer were designed and assembled in school of physics of Madurai Kamaraj University. The only readymade instrument was the digital storage oscilloscope, which was used to make measurements. The block diagram of the so-called 'MADURAI-PA SPECTROMETER' is shown in figure 7. 2.



Block diagram of Madurai PA spectrometer; 1.Source; 2.Monochromator; 3. Chopper; 4. PA cell; 5. Microphone; 6. Pre-amplifier; 7. Lock-in amplifier; 8. Digital Storage Oscilloscope.

Figure 7.2: Schematic Diagram of Madurai – PA Spectrometer

The Photograph of the low cost Madurai- PA spectrometer is shown in figure 7.2a

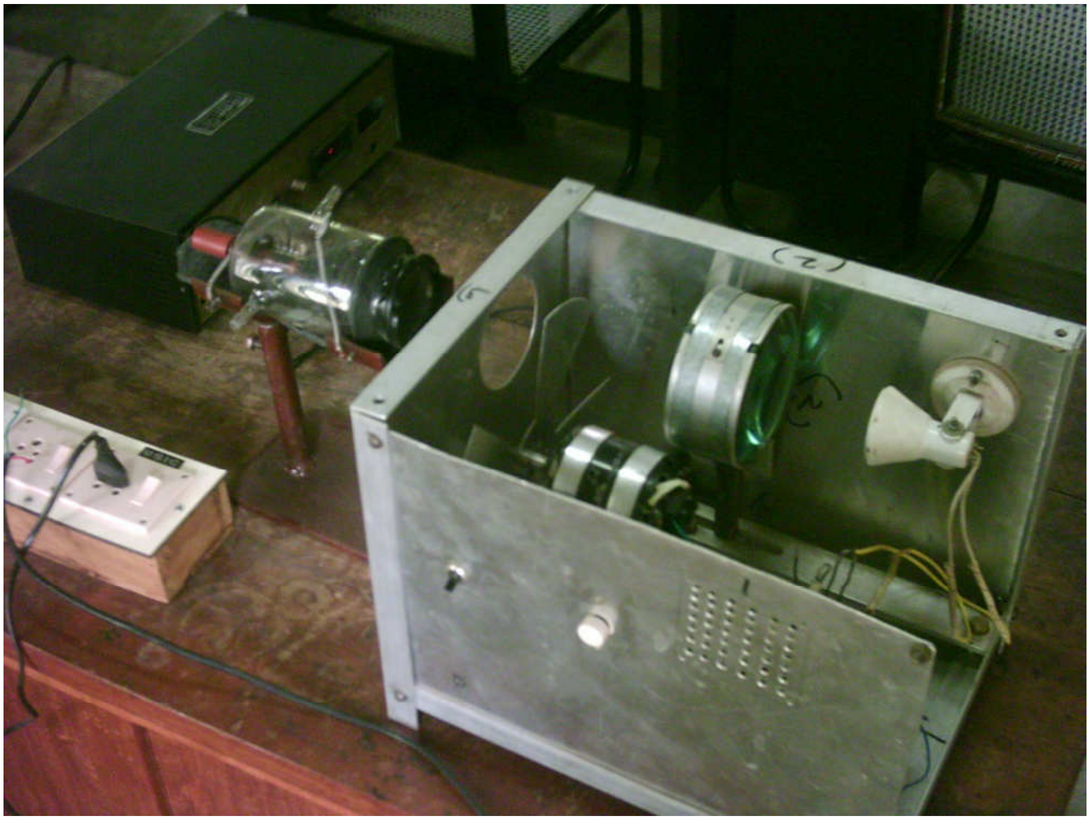


Figure 7.2a: Photograph of Madurai PA Spectrometer

Source

The source is a 1000watt tungsten halogen lamp [2] with the reflecting mirror and condensing lens arrangement, which can be moved, collimated the beam of light back and forth for convenience of focusing. A sturdy transformer was assembled to operate tungsten halogen lamp at 12amperes a.c current and was tested for its performance.

Monochromator

Since the cost of monochromator is high we employed a simple procedure. Since it was intended to carry out the experiments in the visible region of the electromagnetic spectrum, we used colored filter papers (highly transparent) violet, indigo, blue, green, yellow, orange and red. The appropriate filter paper was pasted to a lens holder, which will act as a monochromator when white light passes through it. Care was taken to place the colored filter paper intact and clean.

Chopper

We have made the beam chopper from thick aluminum sheets. The aluminum sheets were cut accordingly so that it will convenient to vary the frequency of the chopper. Hence we cut out three blades of suitable dimensions. The shape of the chopper was that of a fan blade except that the blades are plane instead of twisted. Like this we have designed the different shopper with four blades and two blades. The number of blades increases the frequency of the chopping. This chopping blade with proper arrangements for rotations is connected to an a.c motor. The operating voltage for the a.c motor can be varied from 0-230V. Change in the motor speed changes the chopping frequency. This can be adjusted by changing the power supply voltage, which is variable from 0V to 230V. According to the required experimental frequency a proper chopper blade was selected from the 3 chopping blades. The chopper was placed near or at the focus of the lens. The shape of one of the chopping blades is shown in figure.7.3.

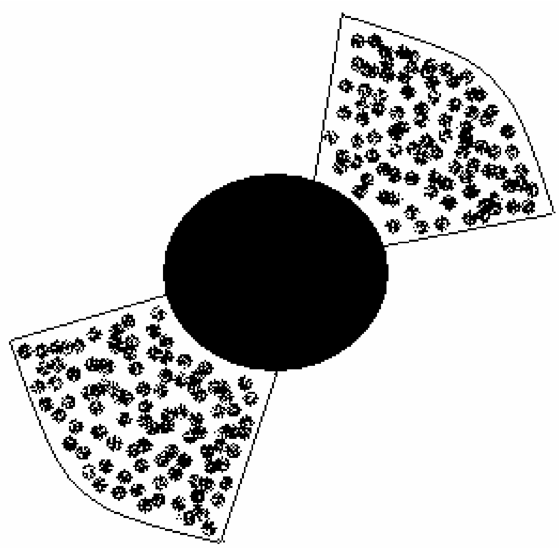
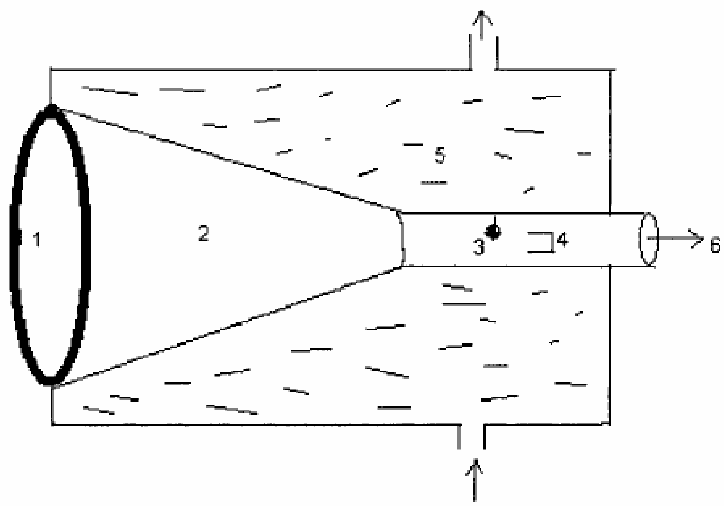


Figure 7.3 Two segment chopper blade

Photoacoustic cell

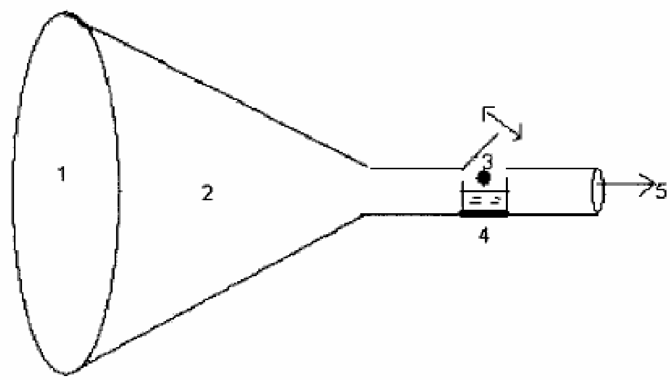
The photoacoustic cell is one of the most important components that require skillful designing suitable for obtaining PA spectra according to the nature of the sample (solid, liquid, gas). For solid samples we have designed a PA cell as shown in figure 7.4.



1. curved watch glass; 2. lampblack coating; 3. microphone; 4. sample holder; 5. thermostat; 6. PA signal to detectors.

Figure 7.4 PA Cell for solid samples

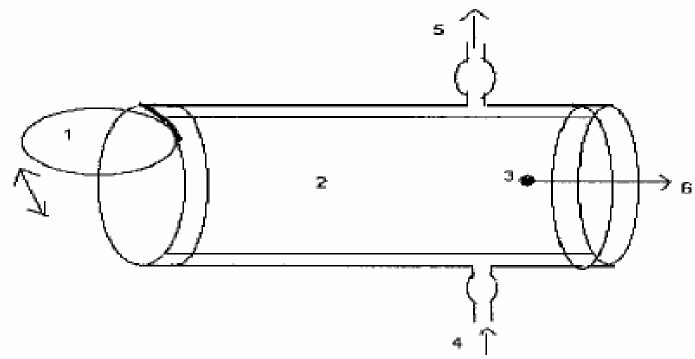
The PA cell is made up of a flint glass funnel whose larger diameter is 10 cm. A funnel of high thermal conductivity material such as metal may result in a weaker signal. Therefore glass funnel was used in our experiment. A cylindrical glass vessel to which liquid can be circulated and the sample in the cell can be kept at any desired temperature by connecting inlet and outlet to the constant temperature bath envelops the whole funnel. The interior of the glass funnel is coated with the lampblack. When opaque layer of carbon has been deposited over the entire interior of the funnel it acts as a backing material. The large opening of a funnel is sealed with watch glass using best adhesive paste. In the curved watch glass the convex side should be placed inside the funnel to reduce volume of the shield chamber and subsequently focusing of the beam. A thin sample holder made of brass/aluminum is placed inside the stem of the glass funnel and in front of the holder a sensitive microphone is placed perpendicular to a sample holder but without touching it. Thin electrical connecting wires are taken from the microphone without disturbing the airtight arrangement of the experimental PA cell setup. One of the detectors namely the microphone is placed inside the funnel which was inevitable. All the other detector devices are assembled in a separate chassis. For liquid samples we have designed a unique PA cell as shown in figure 7.5.



1.curved watch glass; 2. lampblack coating; 3. microphone; 4. quartz sample holder; 5. PA signal to detectors.

Figure 7.5 PA Cell for liquid samples

The PA cell is made up of a small glass funnel. In the center of the stem of the funnel and air tight compartment is drilled to insert a quartz container to hold the liquid sample. The microphone can be placed over the sample without touching it. The interior of the cell is coated with lampblack except the region containing the quartz cell. The entire is made airtight. The large opening of the funnel is sealed with the clean watch glass. For gaseous samples we designed a innovative PA cell as shown in Figure 7.6.



1.quartz glass window; 2. lampblack coating; 3. microphone; 4. thermostat inlet; 5. thermostat outlet; 6. PA signal to detectors.

Figure 7.6 PA Cell for gaseous samples

The cell is cylindrical in shape and there is provision for heating the gaseous sample at any desired temperature by externally circulating liquid system. For PA students of gaseous samples the entire cell should fill the gas and no sample holder is required. The interior of the cell is uniformly coated with lampblack. One of the ends of the cylindrical cell is completely sealed and is blackened inside with lampblack and the microphone is sealed inside the glass vessel. The other end of the cylindrical glass vessel has an opening and a sliding door made of quartz glass will be sealing the entire cell.

Detector

In all the three cells the two terminals of the microphone are projecting outside so that connections can be made easily without disturbing the PA cell. The microphone is connected to an external bias of 2V d.c supply. The other end of the microphone is used as input for the amplifier, which is designed with an IC 741 chip. A very simple and low cost IC 741 operational amplifier with negative feedback is used and the gain is maintained to be 100. This amplifier circuit is shown in figure 7.7.

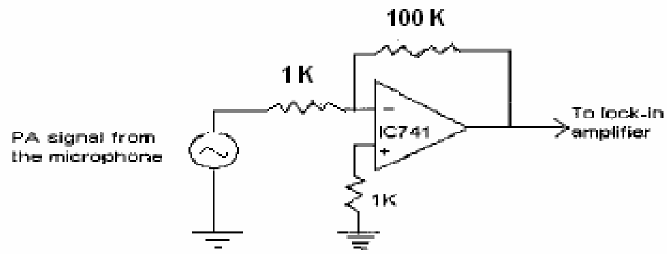


Figure 7.7 Pre-Amplifier Circuit

The power supplies that are needed for this amplifier are constructed with IC 7812 and IC 7912. The lock-in amplifier set up is made p of indigenous electronic components so that it selects only the audio signal, which is at the chopping frequency. A reference signal from the chopper is given to the lock-in amplifier. In the same lock-in amplifier set up which is kept in an iron chassis three digital displays are incorporated to measure chopping frequency, photoacoustic signal amplitude and phase of the photoacoustic signal. Provisions are made to give the output to the digital storage oscilloscope to trace the waveform and to check the PA signal amplitude and phase read by the digital displays are one and the same. In our experiment we used the digital panel meter to find the chopping frequency. The digital storage oscilloscope (DSO, 20MHz, GOULD) measured the PA signal amplitude and phase.

7.2 Results and Discussion

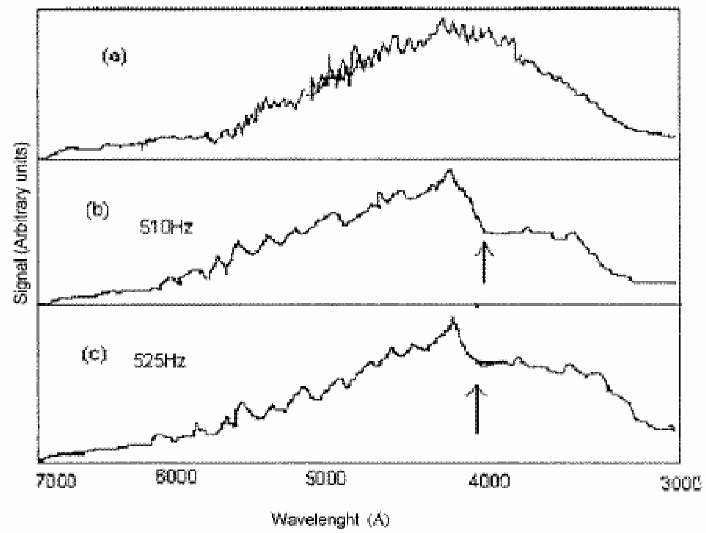
To check the performance of the constructed "MADURAI - PA SPECTROMETER", we conducted experiments with a known solid sample, liquid sample and gaseous sample using the three different cells. The results that were obtained are encouraging and fall within an error of ±3% from the other references. Solid sample By keeping the wavelength fixed (green color filter was used) the depth profile analysis was carried out and the corresponding PA signal is measured by varying the chopping frequency. The sample that was tested was solid poly (methylacrylate) synthesized in our laboratory. The characteristic frequency fc was found out. This frequency is that point at which the sample goes from thermally thin to thermally thick state [3]. The thermal diffusivity of the sample was calculated and found to be 1.9 x $10^{-6}m^2\ sec^{-1}$. Jothi Rajan et al [4] have reported for the same PMA sample with a sophisticated and very costly photoacoustic spectrometer (EG&G MODELS) the diffusivity value of 1.8 x $10^{-6}m^2\ sec^{-1}$. Thus the indigenously designed low cost set up is in no way inferior to the one that was imported.

Liquid Sample

To study the thermal diffusivity of polyaniline in N-methylpyrrolidone (NMP) we used the liquid cell and followed the depth profile analysis and found the characteristic frequency. Hence we found thermal diffusivity to be 25.6 x $10^{-6}m^2sec^{-1}$. Pilla et al [5] have reported the thermal diffusivity value for polyvinylacetate /polyaniline solution by thermal measurements as 1.05 x $10^{-7}m^2\ sec^{-1}$. This measurement is also fairly good and acceptable within the error limits.

Gaseous Sample

We used the gas PA cell to study the photochemical deexcitation of NO_2 gas at a pressure of 10 torr. Here we followed the wavelength scanning method. The chopping frequency is kept fixed and the wavelength is changed and accordingly the PA signal amplitude is measured. Harshbarger and Robin [6] have got a similar type of spectra at a chopping frequency of 510 Hz. In our experiment the chopping frequency was fixed at 525 Hz. The spectra are compared are shown in figure 7.8.



Comparison of the (a) optical and (b) photoacoustic spectra of NO_2 at 10 torr (c) photoacoustic spectra of NO_2 at 10 torr in the present work.

Figure 7.8 Gaseous Spectra

These are in good agreement with our results within the acceptable error limits.

7.3 Digitalized Screw Gauge with Motor Arrangement for Ultrasonic Interferometer (DSMU)

Introduction

Mittal Enterprises, New Delhi, India, supplied the ultrasonic interferometer for velocity measurements. This instrument is used to find the velocity of ultrasound in various liquids and liquid mixtures. This instrument has a screw gauge arrangement, which has a very narrow scale for measurement, and taking readings in the micrometer screw was found very difficult due to diffused light in the laboratory. So to make the measurements easier a digitalized screw gauge motor arrangement was fabricated indigenously making use of microprocessor controlled motor arrangement. The pulse given will rotate the wheel of the motor, which in turn will rotate the micrometer screw. For one pulse the screw gauge moves through 0.0024mm. Therefore, for N pulses the screw would have moved through N x 0.001 mm which gives the screw gauge reading easily and directly. Hence measurements can be made accurately avoiding parallax error and shivering of the hands due to old age can be compensated.

Figure 7.9: Photograph of digitalized micrometer screw gauge with ultrasonic interferometer.

The ultrasonic pulse generator is to generate the pulse using microprocessor IC, transistors and some electronic circuits. Here the speed, count, or rotation is set by using the circuits. The stepper motor will run by specified setting speed. The experimental set up is shown in the photograph (figure7.9). The following components are used in the instrument:

Mains chord, seven segment LED display with circuits, ON/OFF switch, power supply, reset switch, left-right switch, fuse holder, power transistor, regulated transistor, zener diode, microprocessor IC, 7805 regulated IC, 22µf/50V capacitor, diode sets for bridge rectifier, capacitors and resistors, connectors, 4.4V stepping motor and main board circuits.

Working Principle

The working principle of the DSMU is to give the power supply 12v dc power supply to the mains board by using 12 volt transformer and its related circuits. The same voltage is also used for seven segment display circuits.

Here one ON/OFF switch, one left switch, one right switch and reset switch are used. First the speed is set in either left or right side. If the rotation is set in the left side the left switch is pushed to set the count of rotation. The number of counts are displayed in the seven segment display.

At the time the supply voltage goes to the microprocessor IC and transistor. It will control the speed of the speed of the motor to generate the pulse. If the direction is changed the reset switch is used and the right side rotation is set. The microprocessor IC circuits controls and rotates the motor. The stepper motor requires 4 .6v dc . This voltage is taken from the regulated IC7805. Automatically 4.6v goes to the motor. At the same time the microprocessor IC controls left or right side rotation. If the left side rotation is set and to set the speed or count the stepper motor will move at the same time pulse will be generated. If the side is changed to change the number of counts by using reset, then right switch and set the speed or count. The above process is done on the right side. The microprocessor Ic will control either left or right side rotation. In this way the DMSG is working.

7.4 Conclusions

The indigenously and economically designed MADURAI - PA SPECTROMETER will be used for our future studies on nanocomposites of organic and inorganic polymer and biological samples. Since the PA technique is a nondestructive testing and evaluating tool with maximum facilities for obtaining best results of rare samples we intend using it and the same also fits within the limits of our funding. This technique gives fairly accurate results as any other conventional methods. This simple spectrometer can be constructed in college laboratories and number of innovative experiments can be carried out. The total cost of the entire set up came around 900 Euros.

The DSMU was fabricated at a moderate price (US $ 100). This was used by Amirtharaj to study the ultrasonic parameters of N N` Bisacrylamide in water [7]. The usefulness of this device was appreciated and many institutions which where having the manual ultrasonic interferometer purchased the DSMU from the researcher as it was easy to read the measurements.

References

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