CHAPTER 2

Materials and Experimental Techniques

Abstract

In the present research work, different chiral molecules are investigated to observe the optical handedness of chiral materials which are listed in the beginning of the chapter Newton's rings setup, Raman spectroscopy, Raman optical activity (ROA) spectroscopy, and high pressure setup are some of the experimental techniques used to observe differences in the optical handedness of chiral materials. To investigate chiral molecules, plane polarized light and circularly polarized light are the primary requirements. The method to generate them is discussed in this chapter. In the current study, quasi-hydrostatic and non-hydrostatic high pressure studies are performed on chirality-inducing compound, and for characterization purpose, the powder X-ray diffraction (PXRD) technique is employed. This chapter gives details of all different experimental techniques used to carry out present research work.

2.1 Chemicals used in the Research Work

2.1.1 Sample used for Refractive Index Mesurements

1) D Glucose, L-Glucose
2) D-Mannose, L-Mannose
3) R)-(+)-1,1'-Bi-2-naphthol (R BINOL), (S)-(-)-1,1'-Bi-2-naphthol (S BINOL)
4) R)-(+)-alpha methyl benzyl amine, (S)-(-)- alpha methyl benzyl amine
5) (R)-(+)-alpha ethyl benzyl amine, (S)-(-)-alpha ethyl benzyl amine
6) D-Tartaric acid, L-Tartaric acid

2.1.2 High Pressure Raman Spectroscopic Measurements

1) Sodium dithionite ($Na_2S_2O_4$)
2) Acetophenone Extrapure (C_8H_8O)
3) Benzil ($C_{14}H_{10}O_2$)

2.1.3 Raman Optical Activity (ROA) Measurements

1) R-2, 2'- bis (diphenyl phosphino) - 1,1' binaphthyl (R BINAP)

2) S-2, 2'- bis (diphenyl phosphino) - 1,1' binaphthyl (S BINAP)

2.2 Details of Chemicals used throughout the Research Work is shown in Table 2.1.

Table 2.1. Chemicals Details

S.No	Name of Compound	Chemical formula	CAS Number	Purity	Manufacturer
1	D Glucose	$C_6H_{12}O_6$	50-99-7	≥ 99 %	Sigma Aldrich
2	L-Glucose	$C_6H_{12}O_6$	921-60-8	≥ 99 %	Sigma Aldrich
3	D-Mannose	$C_6H_{12}O_6$	3458-28-4	≥ 99 %	Sigma Aldrich
4	L-Mannose	$C_6H_{12}O_6$	10030-80-5	≥ 99 %	Sigma Aldrich
5	R)-(+)-1,1′-Bi-2-naphthol (R BINOL)	$C_{20}H_{14}O_2$	_	_	Synthesised in Lab
6	(S)-(-)-1,1'-Bi-2-naphthol (S BINOL)	$C_{20}H_{14}O_2$	_	_	Synthesised in Lab
7	R)-(+)-alpha-methyl benzylamine	$C_6H_5CH(CH_3)NH_2$	3886-69-9	98 %	Sigma Aldrich
8	(S)-(-)- alpha-methyl benzylamine	$C_6H_5CH(CH_3)NH_2$	2627-86-3	98 %	Sigma Aldrich
9	(R)-(+)-alpha-ethyl benzylamine	$C_6H_5CH(C_2H_5)NH_2$	3082-64-2	≥ 95 %	Fluka
10	(S)-(-)-alpha-ethyl benzylamine	$C_6H_5CH(C_2H_5)NH_2$	3789-59-1	≥ 95 %	Fluka
11	D-Tartaric acid	$C_4H_6O_6$	147-71-7	99 %	Sigma Aldrich

12	L-Tartaric acid	$C_4H_6O_6$	87-69-4	99 %	Sigma Aldrich
13	Sodium dithionite	$Na_2S_2O_4$	7775-14-6	85%	Thermo Fisher Scientific
14	1-phenylethanone (Acetophenone Extrapure)	C_8H_8O	98-86-2	99 %	SDFCL
15	1,2-diphenylethane-1,2-dione (Benzil)	$C_{14}H_{10}O_2$	134-81-6	≥ 99 %	MERCK
14	R-2, 2'- bis (diphenyl phosphino) - 1,1' binaphthyl (R BINAP)	$C_{44}H_{32}P_2$	76189-55-4	≥ 94 %	Sigma Aldrich
15	S-2, 2'- bis (diphenyl phosphino) - 1,1' binaphthyl (S BINAP)	$C_{44}H_{32}P_2$	76189-56-5	≥ 94 %	Sigma Aldrich

2.3 Experimental Techniques

2.3.1 Optical Components to Generate Plane and Circularly Polarized Light

2.3.1.1 Polarizer

A polarizer is an optical component that converts an unpolarized light into plane polarized light. Its transmission axis, also known as the polarization axis, allows to pass electric field components that oscillate in only one plane with respect to the propagation direction. Polarizers are made up of plastic, birefringent material, and glass and are available in the market in different dimensions. Some linear polarizers use the birefringent properties of the material; the Nicol prism is one example of it. It works on the double refraction phenomenon, it absorbs one of the refracted components and allows only one refracted component to pass through it. Sometimes plastic sheet with polyvinyl alcohol (PVA) is heated and stretched so that molecules of PVA are aligned in the direction where the sheet is stretched and then subjected to iodine vapour. Due to this, iodine atoms get attached in the direction where PVA molecules are

aligned. When unpolarized light passes through such a sheet, light in the direction of PVA-iodine molecules is absorbed, and only the perpendicular component is allowed to pass through it. Such Polaroid sheets work as good polarizers. Polarizers have a unique direction known as the transmission axis of the polarizer, along which light vibrations are allowed to pass and rest components of light are obstructed. (Subrahmanyam et al., 2010)

2.3.1.2 Analyzer

Analyzer is an optical component used to analyze the plane of polarization of plane polarized light. The polarizer and analyzer are the same optical components, but they work in different ways. The rotation of the transmission axis of the analyzer with respect to the fixed transmission axis of the polarizer can give maximum, intermediate to minimum intensity depending on the angle between the transmission axes of the two.

2.3.1.3 Quarter Wave Plate (QWP)

A birefringent crystal with its optic axis in the plane of incidence and parallel to its refracting surface constitutes a quarter wave plate. The crystal thickness is cut in a fashion to provide a $\frac{\lambda}{4}$ path difference and a 90˚ phase difference between the e-ray and o-ray. QWP orientation with respect to the polarizer generates circularly and elliptically polarized light. In market, two types of QWPs are available: multi order quarter wave plates and zero order quarter wave plates. For limited range of frequencies multiple order wave plates are used and for wide range of frequencies zero order quarter wave plates are used. (Ghatak, 2017; Subrahmanyam et al., 2010)

Working of Quarter wave plate (QWP)

Due to the birefringence property of the material, double refraction occurs within the material, and either e-ray or o-ray travels faster (slower) than the other within the material. The thickness of a quarter wave plate is kept so that the two refracted rays have a path difference of $\lambda/4$ and a phase difference of $\pi/2$.

The orientation of the quarter wave plate's fast (slow) axis is important because if it is kept at 45˚ with respect to the linear polarizer's transmission axis, it splits the incident ray into two rays, e-ray and o-ray, that are equal in amplitude but have a path difference of $\lambda/4$ and a phase difference of $\pi/2$. When these coherent linearly polarized components of equal amplitudes

combine, circularly polarized light is produced, but if fast (slow) axis is kept at some angle other than 45˚ coherent linearly polarized components are of different amplitudes, and when they combine, elliptically polarized light is produced. (Subrahmanyam et al., 2010)

2.3.2 Photodetector

A photodetector converts optical signal into electrical signal. In present work a 5mm NPN phototransistor (T-1, PT333-3C) made up of photosensitive silicon material acquired from Holmarc Opto Mechatronics Pvt. Ltd. (Model No: HO-ED-PHPD-01) is used. At ambient temperature, the detector's spectral sensitivity ranges from 150 nm to 1200 nm. Photodetector that we used in our experiment is shown in Figure 2.1.

Figure 2.1: Photodetector and Detector output measurement unit.

2.3.3 Prism Table Spectrometer

It is an instrument to analyze the electromagnetic spectrum and different properties of light: frequency, wavelength, and energy. The Prism Table Spectrometer that we used in our present work is shown in Figure 2.2 and was purchased from OSAW (Cat No. 23319). The spectrometer's important parts are the collimator, prism table, and telescope. The collimator consists of a slit, tube and lens. The slit width is adjusted by a knob provided on the side and can limit the amount of incident light. A prism table can be fixed for particular experimental geometry, and a telescope consists of an objective lens and eyepiece.

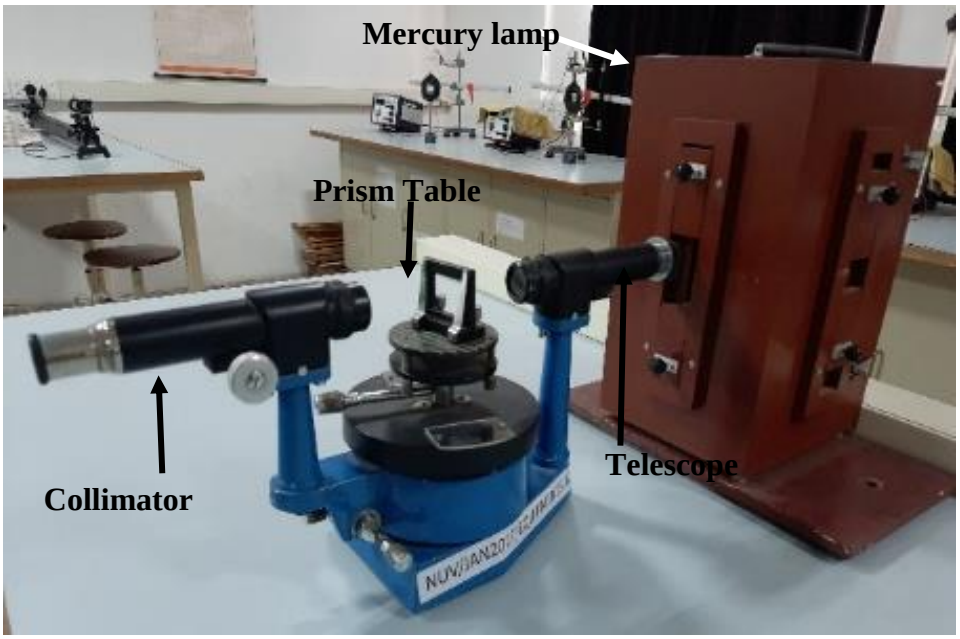


Figure 2.2: Prism Table Spectrometer

2.3.4 Analytical Balance

An analytical balance is used to measure the mass of the sample with high precision and readability up to .00001 grams. The precise measurement of samples is very important in experiments for that analytical balance Uni Bloc (Shimadzu), that we used in our work is shown in Figure 2.3. When samples are measured in an analytical balance, they are kept inside the glass to prevent dust or air from interfering with the measurement.

Figure 2.3: Analytical Balance

2.3.5 Abbe Refractometer

Abbe Refractometer is used to measure refractive index of liquids and work on the principle of critical angle. For this instrument, very less amount of sample (1 to 2 drops) is required for measurement. It consists monochromatic light source for refractive index measurement. Two prisms are present, out of which, lower prism is the illuminating prism with its top surface matted and the upper one is the measuring prism.

Sample (liquid) is filled as a thin layer between these two prisms. When light enters the sample (liquid), it alters the direction of light and light gets refracted from illuminating prism at critical angle and travels parallel to the bottom of the measuring prism along the sample (due to the matted surface of illuminating prism). Light that enters into the measuring prism is a resultant of infinite number of refracted rays from the sample that refracts at all angles. The amount of change in direction of light is refraction. Finally, Rotating knob attached to Abbe refractometer is moved to align the eyepiece with a shadow boundary to distinguish the dark and bright regions. Once alignment is done refractive index value of filled sample is measured. The accuracy of abbe refractometer is ±.0002 and it can measure refractive index in the range of 1.3 to 1.7. (Almicro Abbe Refractometer) Schematic diagram of abbe refractometer is shown in Figure 2.4.

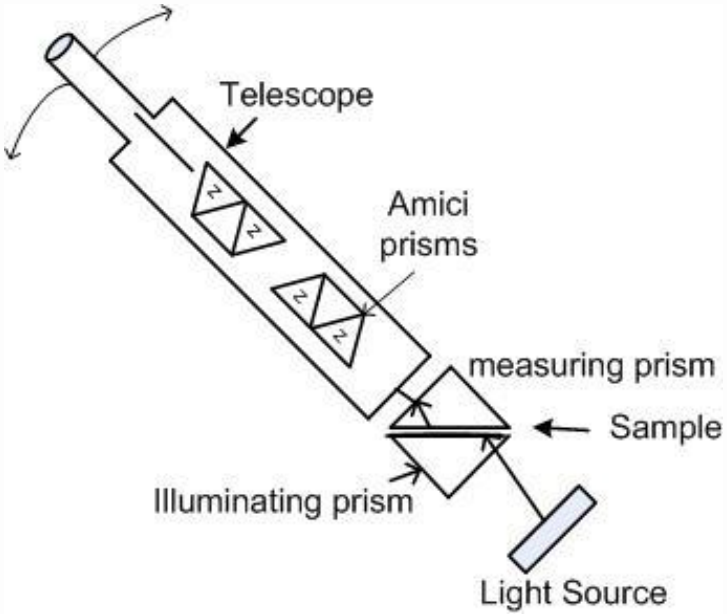


Figure 2.4: Schematic of Abbe Refractometer.

2.3.6 Rudolph J 257 Refractometer

Rudolph Refractometer J 257 is a digital automatic refractometer to measure the refractive index of samples. It can measure refractive index in the range of 1.26-1.72. In it, a sample is placed on the prism and the sample holder is covered. The refractive index of the sample is displayed on a digital screen in seconds. The accuracy of J 257 refractometer is ±.0001.

2.3.7 Polarization Microscope

A Polarizing microscope uses polarized light to illuminate samples and is used to study birefringent samples. A birefringent sample is kept between the polarizer and analyzer, with their transmission axes kept perpendicular to each other. Birefringent materials have the special optical property that their refractive index depends on the polarization and propagation direction of light as they have different densities in different directions. Anisotropic materials are birefringent in nature.

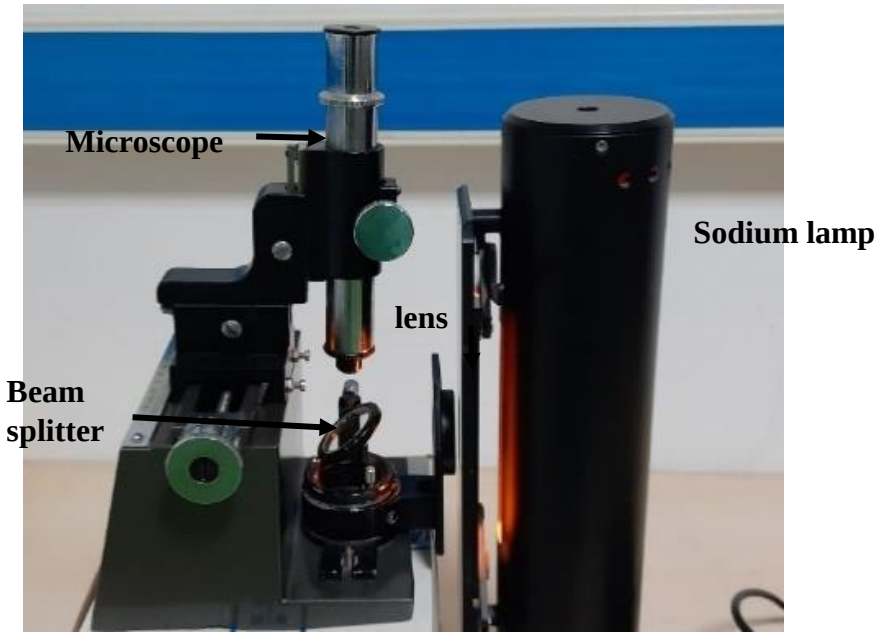
In a Polarizing microscope, polarized light falls on the birefringent sample and gets doubly refracted. The incident beam changes its direction, and some of the light is allowed to pass through the perpendicularly polarized analyzer. Depending on interference, a certain colour is observed, if the orientation, thickness of the birefringent object is changed, the interference colour also changes. Other accessories required in a polarizing microscope are an accessory plate (gypsum), a Bertrand lens, and a condensing lens. To enhance retardation, an accessory plate is used, and Bertrand lenses and a condensing lens are used to find the optic sign of birefringent crystals.. In present thesis work, we tried to find fast and slow axes of quarter wave plate made up of birefringent biaxial crystal mica.(Gribble & Hall, 1993; Nesse, 1991)

2.3.8 Newton's Rings Apparatus

Figure 2.5a shows a typical experimental setup for Newton's rings experiment purchased from OMEGA (Model No: OMEGA TYPE ES-249). L.C of the instrument is .01mm (.001cm) It consists of a Plano-convex lens with a large radius of curvature placed on a plain glass plate and illuminated from the top. A thin circular air film of varying thickness is formed in all directions between the plano-convex lens and the plain glass plate. A specific thickness of air film is present in a circular geometry.Light falls on beam splitter inclined at 45° to the horizontal, and the on the wedge shaped film formed between the plano-convex lens and the

glass plate. Light incident on air film is reflected from the glass to the air boundary and rest is transmitted. The transmitted light is reflected again from the top surface of the glass plate.

Thus, the upper and lower layers of film reflect light incident from the top of the thin film. These rays are coherent because they are produced from the same incident beam through division of amplitude. These reflected rays interfere, and depending on the path difference, bright (constructive) or dark (destructive) circular interference rings are formed. In case of a monochromatic source an interference pattern in the form of alternate dark and bright concentric rings is obtained, but with a polychromatic source, some colourful concentric circular fringes are observed. Interference rings formed with monochromatic sodium light are shown in Figure 2.5b. (Ghatak, 2017; Subrahmanyam et al., 2010) The traditional setup is modified and used for our experiments and the details for the same are mentioned in Chapter 4.



(a)

(b)

Figure 2.5:(a) Newton's Rings Apparatus, (b) Interference rings formed with a monochromatic source (sodium light)

2.3.8.1 Conditions for Constructive and Destructive interference Rings Formation

Conditions for constructive and destructive interference rings are shown in Eq. 1 and in Eq. 2, respectively. Where *x* is the thickness of the film, m is the order of the ring.

$$2x = (2m+1)\frac{\lambda}{2} \tag{1}$$

$$2x = m\lambda \tag{2}$$

2.3.8.2 Diameter of Interference Rings

To calculate diameter of interference ring schematic arrangement is drawn in Figure 2.6. Let the plano-convex lens of large radius of curvature R is placed on plain glass plate, Let the thickness of air film is be and is diameter of dark ring is 2p.

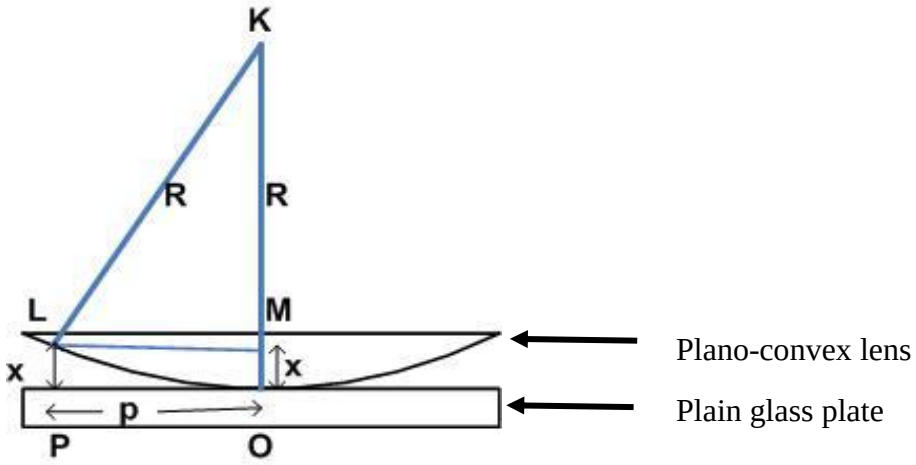


Figure 2.6: Schematic showing geometrical details of plano convex lens and plane glass plate.

Applying Pythagoras theorem:(Subrahmanyam et al., 2010)

$$R^2 = p^2 + (R - x)^2 \tag{3}$$

x is small compare to R, neglecting x^2, Eq. 3 can be written as

$$p^2 = 2Rx \tag{4}$$

Applying condition of minima (Eq. 2) and substituting 2x value in Eq. 4.

$$p = \sqrt{m\lambda R} \tag{5}$$

Since p is the radius of dark ring (Eq. 5), the diameter of the ring is twice of p.

$$D = 2p = 2\sqrt{m\lambda R} \tag{6}$$

Diameter of dark ring is measured with Eq. 6. Where m is the number of the ring from the center, λ is the wavelength of the incident light and R is the radius of curvature of plano-convex lens.

2.3.8.3 Refractive Index Measurement

When a drop of liquid sample is filled in the cavity between the glass plate and the plano-convex lens, a thin wedge shaped film of liquid sample is formed between them. Now the conditions of maxima and minima are modified due to the refractive index of the liquid sample. Let μ is the refractive index of liquid sample. the condition for maxima will be modified as shown in Eq. 7:

$$2\mu x = (2m+1)\frac{\lambda}{2} \tag{7}$$

The condition for minima will be modified as shown in Eq. 8:

$$2\mu x = m\lambda \tag{8}$$

Substituting value of x from Eq. 4 in Eq. 8.

$$2\mu\left(\frac{p^2}{2R}\right) = m\lambda$$

$$D^2 = \frac{4m\lambda R}{\mu}$$

Let diameter of m^{th} ring and $(m+q)^{th}$ ring of liquid thin film is

$$D^2_{m(liq)} = \frac{4m\lambda R}{\mu}$$

$$D^2_{m+q(liq)} = \frac{4(m+q)\lambda R}{\mu}$$

$$D^2_{m+q(liq)} - D^2_{m(liq)} = \frac{4q\lambda R}{\mu} \tag{9}$$

For air film:

$$D^2_{m+q(air)} - D^2_{m(air)} = 4q\lambda R \tag{10}$$

Dividing Eq. 10 by Eq. 9, we can calculate the refractive index (μ) of the liquid filled between plain glass plate and plano convex lens from Eq. 11. (Subrahmanyam et al., 2010)

$$\mu = \frac{D^2_{m+q(air)} - D^2_{m(air)}}{D^2_{m+q(liq)} - D^2_{m(liq)}} \tag{11}$$

2.3.9 Circular Dichroism (CD) Spectroscopy

Circular dichroism spectroscopy is a very sensitive technique based on optical absorption and is mainly used to study optically active chiral molecules to investigate their structural details.

Circularly polarized light of different handedness: right circularly polarized light (RCP) and left circularly polarized light (LCP) interacts with the molecule molecule under investigation alternately. The difference in absorption of RCP and LCP light at certain wavelengths gives structural details of the chiral molecule. (Berova et al., 2000; Sato, 2020; Snatzke, 1968; W.Woody, 1995) Schematic of Circular dichroism spectroscopy is shown in Figure 2.7.

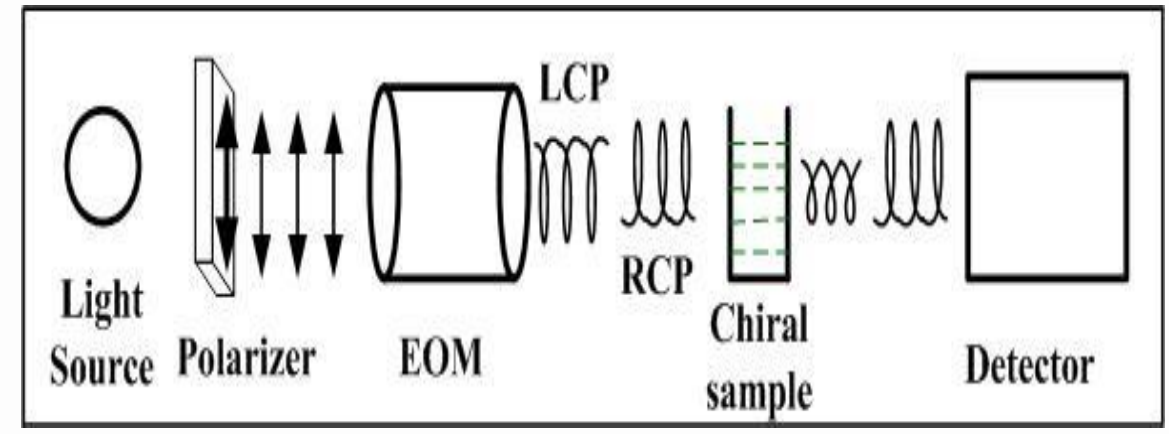


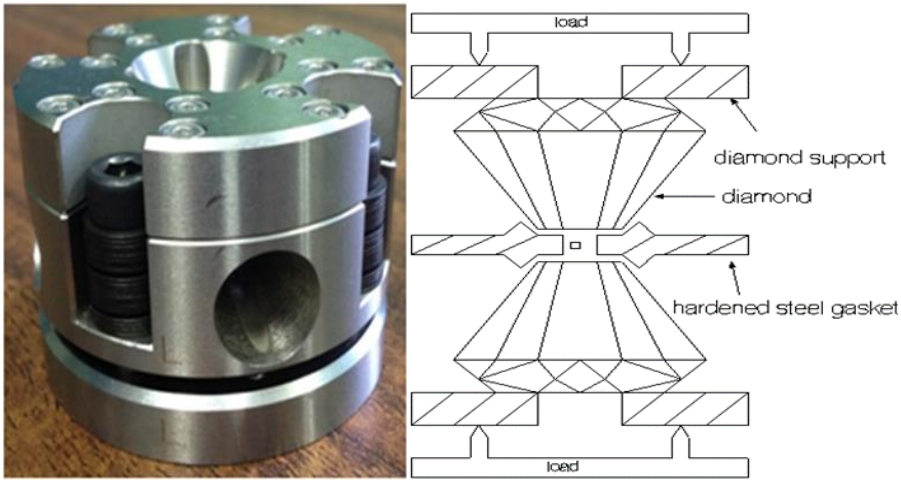
Figure 2.7: Schematic of Circular Dichroism Spectroscopy.

2.3.10 High Pressure Technique

Diamond Anvil Cell (DAC)

For high pressure experiments, a diamond anvil cell (DAC) and stainless steel gasket are required. Compression is done in DAC between a pair of Type I diamonds to hold the sample, a pre-indented stainless steel gasket is used. The DAC loaded with sample is kept in the light path and *in-situ* data is collected.

For our experiment, Syntek symmetric DAC shown in Figure 2.8, which has Type I diamonds with a culet size of 400 microns was used. The stainless-steel gasket (T301) is pre-indented to a specific thickness of 40-50 μm and has a centrally drilled hole of a size 120 μm.



(a) (b)

Figure 2.8 (a) Syntek symmetric diamond anvil cell (DAC), (b) Schematic arrangement of pair of diamonds, inside the cell.

Experiments in DAC can be performed in quasi-hydrostatic, hydrostatic, and non-hydrostatic pressure environments. To generate quasi-hydrostatic pressure and hydrostatic pressure, along with sample pressure transmitting medium needs to be filled. While no medium is required in non-hydrostatic pressure experiments, only the sample needs to be filled in the sample cavity.

For pressure measurement, fine ruby powder is used. Ruby consists of two separate fluorescence peaks, R_1 and R2, and pressure calibration is done with the R_1 ruby fluorescence line. Fine ruby powder is placed in the sample cavity along with the sample and pressure is calibrated with wavelength shift (nm), as shown in Figure 2.9. Ruby calibration equation for Quasi-hydrostatic and non-hydrostatic environment is shown in Eq. 12.

$$\boldsymbol{P = \frac{A}{B}\left\{\left[1 + \left(\frac{\Delta\lambda}{\lambda_0}\right)\right]^{B} - 1\right\}} \qquad 12)$$

This calibration relation shown in Eq. 12 measure the high pressures shift of the ruby R_1 line, where P stands for the pressure in megabars, and λ is the wavelength of the Ruby R line. For a quasi-hydrostatic environment, A=19.04 Mbar, B=7.665, and for a non-hydrostatic environment, A = 19.04, B = 5. (Mao et al., 1986)

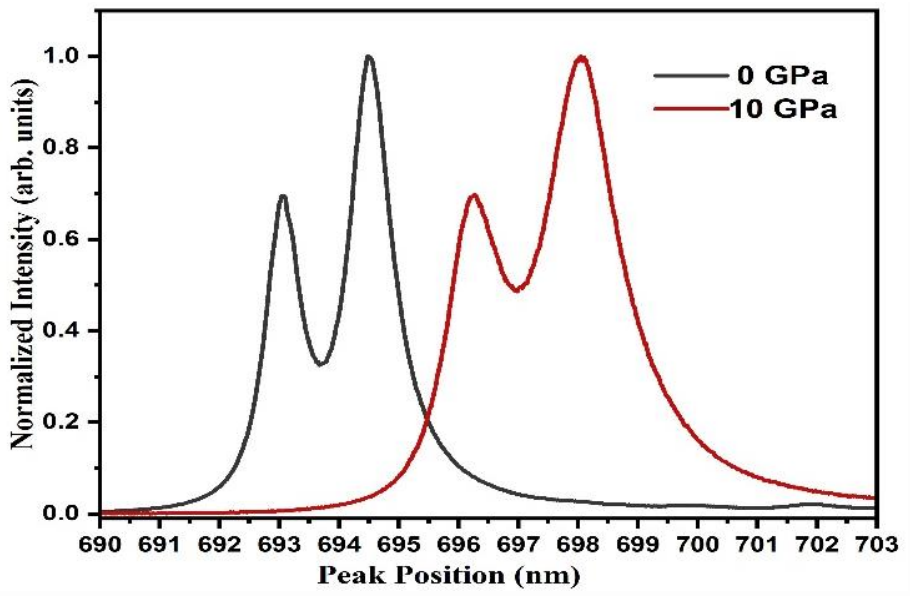


Figure 2.9: Pressure calibration with ruby fluorescence.

2.3.11 Stereo Zoom Microscope

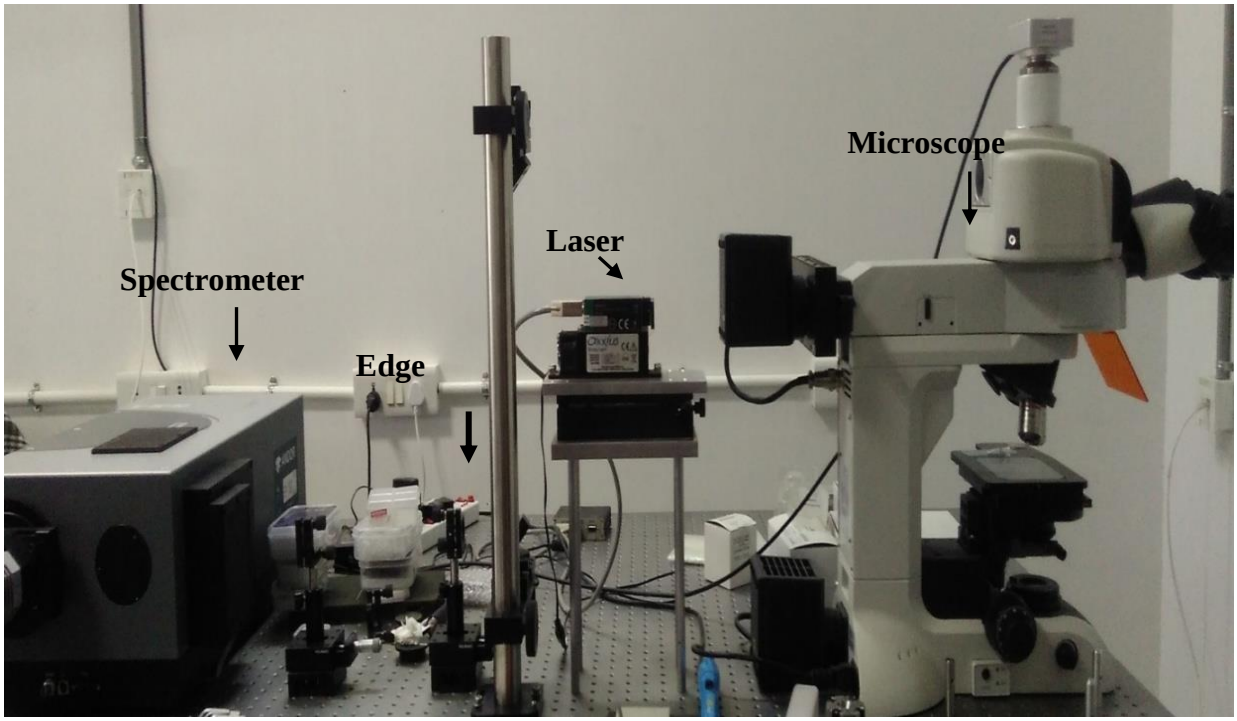
A stereo zoom microscope (Nikon SMZ 1000) used in present work shown in Figure 2.10. It is a powerful, advanced, customizable microscope with a zoom range of 0.8x - 8x and high magnification. It has binocular head attached with CMOS digital camera (Vue) and a computer. Broad wavelength light sources are used in reflection as well as transmission geometry. The stereo microscope gives a very good quality depth dependent images and was used to load samples inside DAC.

Figure 2.10**:** Stereo Zoom Microscope

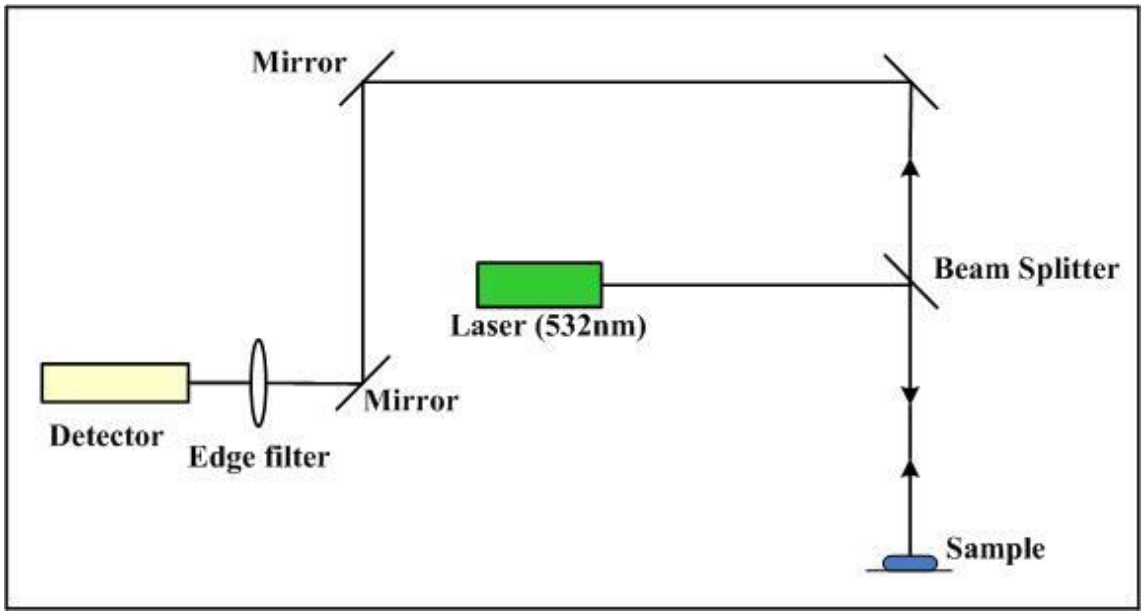
2.3.12 Raman Spectroscopy

Raman spectroscopy a powerful spectroscopic technique based on scattering of light. Details of the technique are discussed in Chapter 1. (Banwell & Mccash, 2017) In the present resarch work, Raman spectroscopy was used to study chirality inducing compounds. Detailed structural characterization was done at high pressure (extreme conditions) in quasi hydrostatic and in non-hydrostatic pressure environment.

In this thesis work, to record Raman spectra 532 nm single frequency Oxxius laser (model LCX-532S-100-CSB-PPF), France was used as an excitation source. Raman spectra is collected in 180˚ backscattering geometry. The Raman spectra were measured using a Spectrometer model: SR-500i-A-R with an 1800 lines/mm grating. Spectrometer is attached to a thermoelectrically cooled Andor iDUS charge coupled device camera. Motorized microscope -Microscope-ELV-100-ND is used in the experiment. Raman spectrometer used in our experiment is shown in Figure 2.11a. Raman Spectrometer schematic representation is done in Figure 2.11b. We performed experiment from the wavelength range from 105 - 4304 cm^{-1} with spectral resolution of 0.13 nm or 4.3 cm^{-1} with 1800lines/mm grating, 26 micron x 26 micron size pixel and entrance slit width 100 micron.



(a)



(b)

Figure 2.11:(a) Raman Spectrometer, (b) Raman Spectrometer Schematic representation.

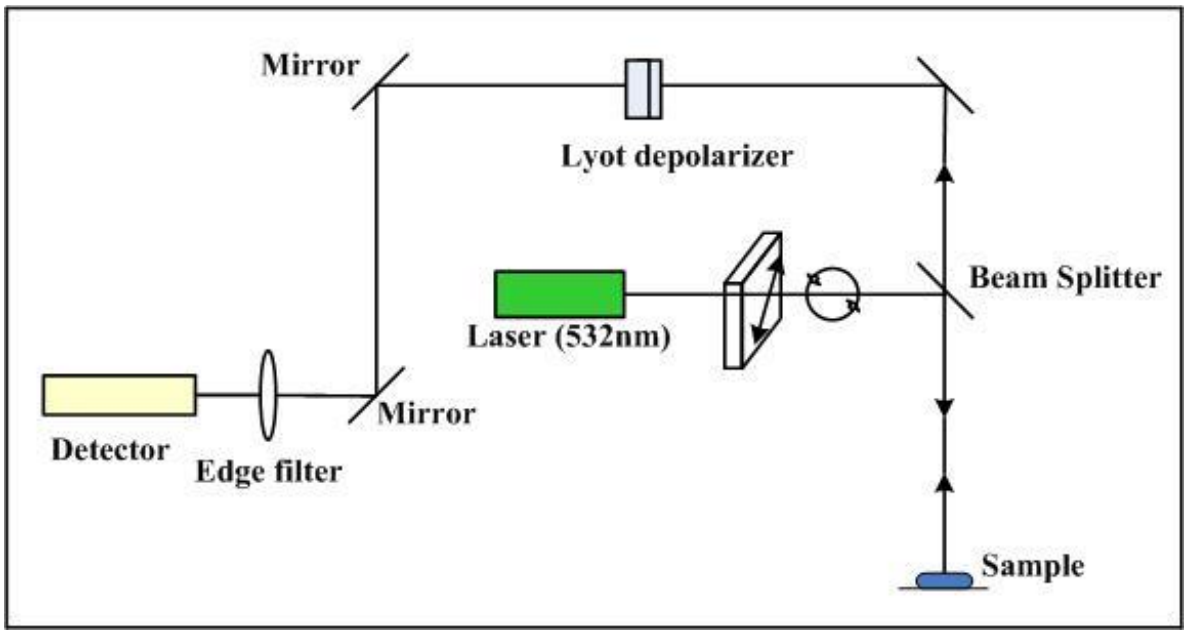
2.3.13 Raman Optical Activity (ROA) Spectroscopy

Raman optical activity (ROA) is a unique spectroscopic technique that analyses the difference in Raman scattering intensities of left and right circularly polarized light from chiral samples and offers extensive stereochemical, vibrational, and absolute configurational information about the chiral molecule. (Yamamoto & Watarai, 2010) (Lightner et al., 2021). The ROA interference mechanism depends on the chiral molecule's polarizability and optical activity tensors. (Barron et al., 2007; Hecht & Barron, 1994) Detailed description of ROA is discussed in Chapter 1. BioTools, Inc. manufactures the Chiral*RAMAN* equipment, which is based on scattered circular polarization (SCP).(Hug, 2006; Hug & Hangartner, 1999) When linearly polarized or unpolarized light hits the sample, the difference in scattered light comprising right and left circularly polarized light is measured. ROA spectra is able to differentiate molecular structure of both enantiomers.

Figure 2.12a depicts the schematic of Raman optical activity (ROA) setup used in this study and Figure 2.12b is the modified Raman optical activity (ROA) on which we conducted experiments in our lab. We recorded Raman optical activity spectra of multiple chiral samples of both enantiomers using Raman spectrometer as per the details mentioned in section 2.2(k).

To generate right and left circularly polarized light zero order quarter wave plate (532nm) is used. Its fast axis is oriented manually +45˚ and -45˚ to generate right and left circularly polarized light respectively. Lyot Depolarizer (model. no. HO-LDP-10), size (10mm x 10mm x 6mm), dimensional tolerance of +/-0.2mm, made up of quartz, wavelength range 200-2200nm is used to remove any polarization component present in the scattered signal.

In the present work, ROA spectra were recorded for both enantiomers of chiral sample under high pressure in non-hydrostatic environment. We performed experiment from the wavelength range from 63 -3170 cm^{-1} with spectral resolution of 0.13 nm or 4.3 cm^{-1} with 1800lines /mm grating, 26 micron x 26 micron size pixel and entrance slit width 100 micron. To collect high quality data, we accumulated data in four separate windows for 300 s x 2 for each window for each enantiomer.



(a)

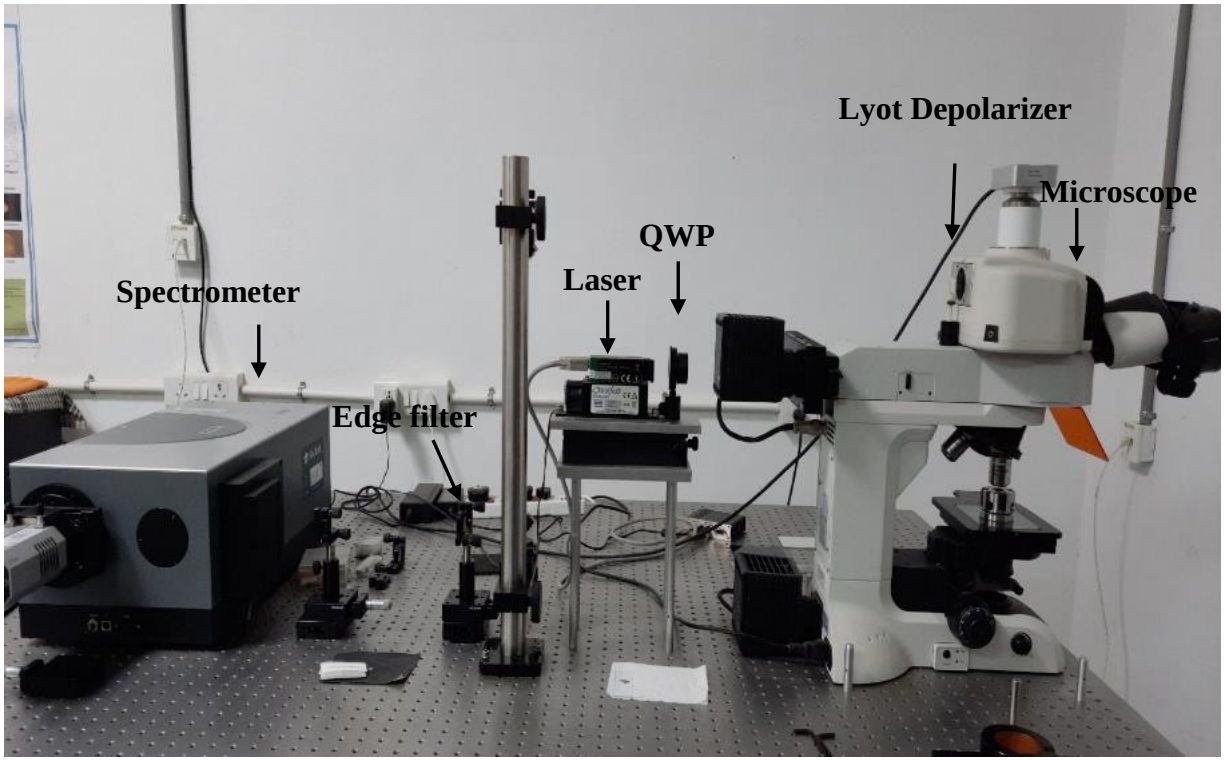


Figure 2.12: (a) Schematic representation of Raman optical activity setup. (b) Modified Raman Optical Activity setup.

2.3.14 Infrared Spectroscopy

It is an absorption spectroscopy used to characterize materials, but in the present work, we used an infrared spectrometer to find the fast and slow axes of a quarter wave plate (589.3 nm; multiple order) by placing it between a polarizer and analyzer with their axes kept parallel. In order to determine the fast and slow axes of a quarter wave plate, we tried to find the difference in transmittance between both the axes. Infrared spectrometers consist of an infrared source. Infrared radiation travels with minimal absorption through the fast axis and with maximum absorption through the slow axis as shown in Figure 2.13. These alternate transmittance values are correlated with the fast and slow axes of the quarter wave plate.(Guohua et al., 1990)

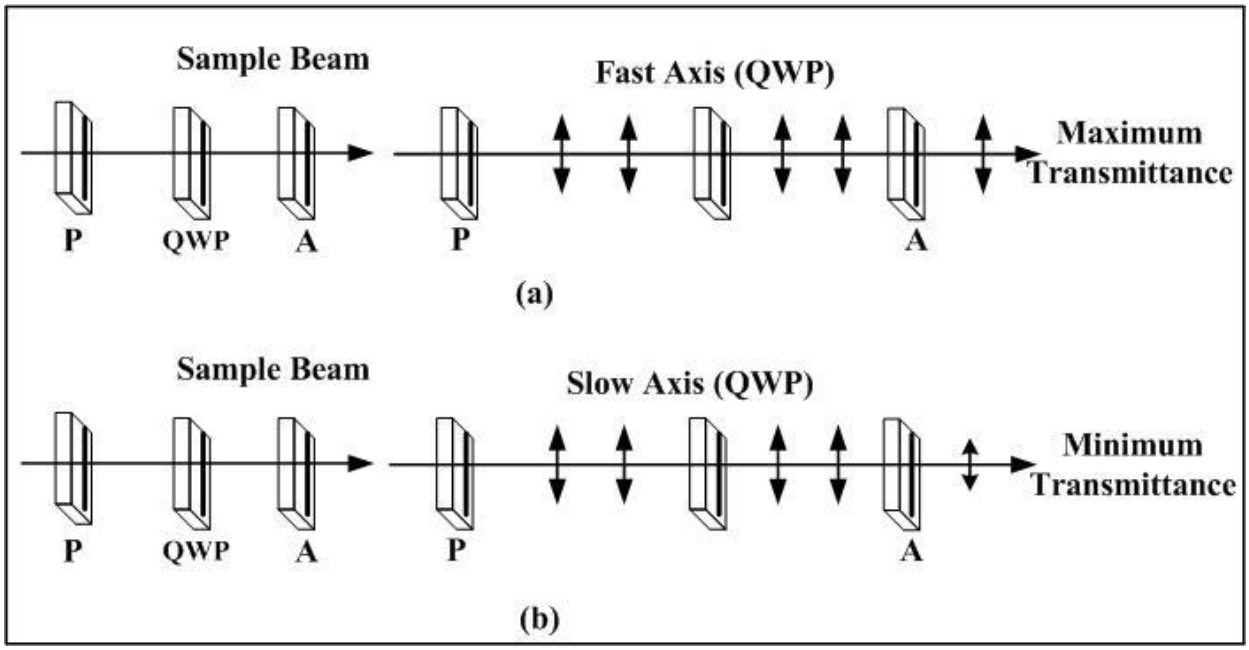


Figure 2.13: Transmission of IR radiation through fast and slow axis.

2.3.15 Powder X-ray Diffraction (PXRD)

Powder X-ray diffraction is a powerful characterization technique that investigates materials in their powder form. The measurement requires less sample preparation. It provides immediate and extensive information about the material, such as structure, phase identification, sample purity, and the morphology of the material. (Holder & Schaak, 2019) Different materials exhibit different diffraction patterns which are used to refine and identify lattice parameters.

In the current work, the powder PXRD measurements were performed using Bruker Model D8 Advance A25, in continuous scan mode at room temperature from 10˚ to 80˚ with a step size of .010. Data was collected by the LynxEye detector. Sample is smeared on an amorphous silica holder and fixed on the sample stage on the goniometer. The instrument is set with B-B geometry. The current and voltage are set to 40 mV and 40 mA, respectively.

2.3.16 Softwares used for Calculations

2.3.16.1 Origin 8

Origin 8 is a software program developed by OriginLab Corporation, USA(1992). It is a very useful tool for creating scientific graphs and analysing data. The software was used multiple

times in our research to plot graphs, analyse data, and for baseline correction, peak fitting, and peak analysis.

2.3.16.2 Crysfire Powder Diffraction Indexing Software

Crysfire, an automatic powder diffraction indexing system program provides fast indexing. Software was used in our research work to identify the crystal system and assign point group..

2.3.16.3 Checkcell

Checkcell is a software used to assign space group from the PXRD data. It computes the "best solution" for a particular cell by comparing observed and calculated peaks.