Chapter II

Experimental Techniques
2.1 Introduction

The investigations on the physical, chemical and mechanical properties of crystals are very important in view of their applications in science and technology. The sophistication in the instrumentation technology offers excellent tools for their characterization. This research work involves in the synthesis, growth of NLO single crystals by slow evaporation technique and the investigations of their structural, thermal, mechanical and optical properties. This chapter describes the experimental methods adopted for the synthesis, crystal growth and characterization of the materials reported in this thesis.

2.2 Crystal Growth Techniques

Growth of single crystals is a challenging task, and the techniques followed for crystal growth aspects differ from crystal to crystal depending on their physical and chemical properties such as solubility, melting point, decomposition, phase changes etc. Moreover, it differs in accordance with the quality and quantity of the starting material and the
perfectness of the crystals in addition to the period and economy. Research on the growth of single crystals has been developed over the years to meet the needs of modern technology. Basic methods have been modified and refined to enhance their applicability. The aim of the crystal grower is to prepare crystals of the best possible quality and quickly with required specifications. Crystal growth methods can be broadly classified on the basis of the types of phase transition during the growth as [1,2]

- Solid growth $\rightarrow$ solid – solid phase transition
- Solution growth $\rightarrow$ liquid – solid phase transition
- Vapour growth $\rightarrow$ Vapour – solid phase transition

Crystal growth from liquid falls into four categories namely, melt, flux, hydrothermal and low temperature growth methods. In the present investigation, low temperature solution growth technique is followed for crystal growth. Hence, a brief account of the solution growth method and its merits and demerits are discussed below.

### 2.3 Crystal Growth From Solution

Among the various methods followed for crystal growth, solution growth at low temperature occupies a prominent place in view of its versatility and simplicity. The solution growth technique yields good
quality single crystals for variety of applications such as electro-optic modulators, piezoelectric oscillators and nonlinear optical applications such as fibre optic communication, optical data storage devices, optical frequency converters etc. [3,4]. Solution growth technique has many advantages than the other methods. In this technique, since the growth is carried out at ambient temperature, the structural imperfection is relatively low. This method is suited for materials having moderate to high solubility in the temperature range from ambient to 100°C at atmospheric pressure and those which decompose on heating and those materials which exhibit structural transformation while cooling.

2.4 Selection of Solvents

A good solvent ideally displays the following characteristics:

(i) A good solubility (about 30-40 gms/100 ml)
(ii) A small vapour pressure
(iii) Low viscosity
(iv) Non-corrosiveness
(v) Non-toxicity
(vi) Non-flammability and
(vii) Low price in pure state

It is known that the solvents provide some control over the crystal habit and this effect depends on the interaction of the surface of the
crystal and the solvent molecules as the crystal grows from the solution. Solvents in practical use include water (H₂O, D₂O), ethyl alcohol, acetone, carbon tetrachloride, hexane, xylene and many others. Almost, all the crystals produced by the low temperature solution growth are grown from water. This is because water has all the properties of a good solvent mentioned above and is available enormously. For crystal growth, high purity water is needed and so double or triple distilled water is used for the synthesis and crystal growth process.

2.5 Solubility

The solubility of a material in a solvent decides the amount of the material, which is available for the growth and hence defines the size of the crystal that can be grown. The solubility of the solute can be determined by dissolving the solute in the solvent maintained at constant temperature with continuous stirring using a magnetic stirrer. On reaching saturation, the equilibrium concentration of the solute can be determined. A sample of the supersaturated liquid is withdrawn by means of a pipette and gravimetrically analyzed. On repeating the above procedure for different temperatures, the solubility curve can be plotted. A detailed investigation of the relationship between supersaturation and spontaneous crystallization was reported by Meirs and Isaac [5]. These results are represented in the solubility diagram.
(Fig. 2.1). Here, the solubility – concentration region is divided into three zones viz., I, II and III respectively. Region I corresponds to the unsaturated zone, where the crystallization is not possible. The region II between the super solubility curve and the solubility curve is termed as metastable zone. Spontaneous nucleation is not possible in this region and hence, a seed crystal is essential to facilitate crystal growth. In the third region, there is an unstable or labile zone where, the spontaneous nucleation is probable.

Fig 2.1 Meir’s solubility diagram.

2.6 Constant Temperature Bath (CTB)

Since the temperature affects the driving force of crystallization (approximated by Gibbs free energy difference between supercooled liquid and crystal) very much, highly stable temperature maintenance is essential throughout the growth process. To achieve the stable
temperature, an active thermostating system to control the temperature to a high degree of accuracy is necessary. The general design of a thermostat with a control system known as constant temperature bath (CTB) includes a thick walled glass chamber filled with water, heating element, temperature sensor, control relay, temperature indicator, stirrer and illuminating lamp. In long periods necessary to grow crystals and the need to avoid any interruption of the temperature control process requires an uninterrupted power supply. In the CTB, the power to this lamp is controlled by a triac based electronic circuit comprising active and passive components. For initial heating an immersion heater of 500 W is fixed at the bottom of the chamber. The system uses a contact thermometer for temperature control. The CTB has provision to set and read the temperature with an accuracy of ±1°C in the temperature range from ambient to 100°C.

2.7 Slow Evaporation Technique

This is the best method to grow good quality single crystals from solution. The temperature is kept constant, and provision is made for slow evaporation. With nontoxic solvents like water, it is permissible to allow evaporation into the atmosphere. The evaporation technique of crystal growth has the advantage that the crystals grow at a fixed temperature. In contrast to the cooling method in which the total mass
of the system remains constant. In the solvent evaporation method, the solution loses particles, which are weakly bound to other components, and therefore, the volume of the solution decreases. The vapour pressure of the solvent above the solution is higher than the vapour pressure of the solute and therefore, the solvent evaporates more rapidly and the solution becomes supersaturated.

2.8 Characterization Techniques

2.8.1 Structural Studies

The structural characterization in the analysis of any material is very important to confirm the synthesized material for its formation. Structural characterization techniques include the following:

(i) Elemental (CHN) analysis to estimate the percentage composition of carbon, hydrogen and nitrogen in the synthesized materials.

(ii) Powder X-ray diffraction to identify the compounds.

(iii) Single crystal X-ray diffraction to measure the unit cell dimensions and morphology of the grown crystals.

(iv) FT-IR spectral analysis for the identification of the functional groups present in the molecules (molecular structure).

(v) $^1$H and $^{13}$C-NMR spectral analyses to study the carbon-hydrogen network of the organic and semiorganic materials.
2.8.2 Mechanical Study

(i) Microhardness for the grown crystals

2.8.3 Thermal Analyses

(i) Melting point.

(ii) Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) for thermal stability of the materials.

(iii) Differential scanning calorimetry (DSC) analysis to evaluate the specific heat of the materials.

2.8.4 Optical Studies

The following linear and nonlinear optical characterizations have been performed:

(i) UV-Vis- optical transparency window

(ii) Refractive index – Brewster’s angle technique

(iii) Birefringence - Interference technique

(iv) The powder SHG efficiency - Kurtz and Perry technique

(v) Laser damage threshold for the grown crystals – surface stability.
2.9 Basic Principles and Instrumentation

2.9.1 Single Crystal X-ray Diffraction

The molecular structure, bond length, bond angle, molecular orientation and packing of molecules in single crystals can be determined by X-ray crystallography [6].

The monochromatic X-rays incident on a plane of a single crystal at an angle $\theta$ are diffracted according to the Bragg’s law

$$2d \sin \theta = n\lambda$$  \hspace{1cm} (2.1)

where $d$ is the interplanar spacing of the incident plane, $\lambda$ is the wavelength of X-ray and $n$ is the order of diffraction. The intensity of the diffracted rays depends on the arrangement and nature of the atoms, ions or molecules in the crystal. Collection of intensities on a full set of planes in the crystal contains the complete structural information about the molecule. Fourier Transformation techniques are used to determine the exact coordinates of atoms in the unit cell from this data.

The Enraf Nonius CAD4 single crystal X-ray diffractometer used for intensity data collection in the present investigations is a fully automated four circle instrument controlled by a computer. It consists of a goniometer, CAD4 interface and equipped with a printer and a plotter. The detector is a scintillation counter. A single crystal is mounted on a thin glass fibre fixed on a goniometer head. The unit cell dimensions and orientation matrix are determined using 25 reflections and then the
intensity data of a given set of reflections are collected automatically by the computer. The ideal dimensions of the single crystal required are approximately \(0.3 \times 0.3 \times 0.3\) mm\(^3\). However, these dimensions may vary based on the habit of the crystal growth and the diffracted beam intensity. The complete three-dimensional crystal structure of the new compounds was solved using a Stoe IPDS II two-circle X-ray diffractometer.

2.9.2 Infrared (IR) Spectroscopy

IR spectroscopy is one of the most widely used tools for the detection of functional groups in pure compounds and mixtures, and for the comparison of compounds. IR region of the electromagnetic spectrum extends from 20 to 4000 cm\(^{-1}\).

There are two types of IR spectrometers characterized by the manner in which IR frequencies are handled. In the first type called the dispersive type, the IR light is dispersed into individual frequencies using a grating monochromator whereas in the second called the Fourier Transform IR where, the IR frequencies interact to produce an interference pattern and this pattern is then analyzed mathematically using Fourier Transform to determine the individual frequencies and their intensities \([7,8]\).
Fig. 2.2 Block diagram of modern FT-IR spectrometer.

Fig. 2.2 shows the block diagram of a modern FT-IR spectrometer. In this spectrometer, the IR light from a suitable source passes through a scanning Michelson Interferometer (Fig. 2.3) and the Fourier Transform gives a plot of intensity Vs frequency. When a sample is placed in the path of the beam, it absorbs the characteristic frequencies so that their intensities are reduced in the interferogram and the ensuing Fourier Transform is the infrared absorption spectrum of the sample. The scan time for the moving mirror dictates the speed with which the IR spectrum can be recorded. Digitalization of the data and calculation of the Fourier Transform take a few seconds more, but the information which constitutes the spectrum, can be acquired in exceedingly short times even in a few milliseconds. Slower scans allow the accumulation of more intense signals, and signal to noise ratio of $10^5$:1 can be reached.
The FT-IR spectra of the synthesized materials were recorded using a Jasco 460 Plus FT-IR spectrometer in the range 400-4000 cm$^{-1}$ by KBr pellet technique.

![Michelson interferometer](image)

**Fig.2.3** Michelson interferometer used in the FT-IR spectrometer.

### 2.9.3 Nuclear Magnetic Resonance (NMR) Spectroscopy

NMR spectroscopy is one of the powerful techniques to confirm the molecular structure of the synthesized compounds. At first, it was developed to study the nuclei such as $^1$H, $^{13}$C, $^{19}$F and $^{31}$P with different spin states. Later on, this technique was used to determine the structure of the organic compounds.
If an electromagnetic radiation of the appropriate energy is applied to nuclei that have been oriented by a magnetic field, a nucleus in the $\alpha$-spin state will absorb the radiation flipping its spin and entering the $\beta$-spin state. The energy absorbed in this transition is equal to the
energy difference $\Delta E$ between the $\alpha$ and $\beta$-spin states, shown in Fig.2.4. The transition is governed by the resonance condition

$$\Delta E = h\nu = h \left(\frac{\gamma}{2\pi}\right) H_0$$

(2.1)

where $\gamma$ is the gyromagnetic ratio (the ratio of the magnetic moment of a rotating charged particle to its angular momentum) [9]. Since each nucleus has its own gyromagnetic ratio, different energy sources are required to bring different kinds of nuclei into resonance in a spectrometer with a given operating frequency. The operating frequency depends on the strength of the applied magnetic field. Modern spectrometers are equipped with radiation sources that can be tuned to different frequencies so that they can be used to obtain the NMR spectra of different kinds of nuclei.

In pulsed FT-NMR spectrometers, a radio frequency pulse of short duration excites all the nuclei simultaneously, and all the signals are collected at the same time using a computer. Many hundreds of individual runs are collected and the signals are averaged. The data are mathematically converted using Fourier Transform and the spectrum is obtained. This method reduces the noise almost to zero (since background noise is random, it averages close to zero). FT-NMR is much faster and more sensitive than CW- NMR. Consequently, spectra can be obtained with less than 5 mg of the compound.
In the present study, $^1$H and $^{13}$C – NMR spectra of the synthesized samples were recorded using a JEOL GSX 400 MHz FT-NMR spectrometer.

2.9.4 Vicker’s Microhardness Tester

Physically, hardness is the resistance offered by a solid to the movement of dislocations. Practically, hardness is a resistance offered by a material to localized plastic deformation caused by scratching or by indentation. The indentation hardness is measured as the ratio of the applied load to the surface area of the indentation. In principle, the indentation hardness measurement can be carried out at fairly high loads (~100 kg). But for materials which have low hardness and small size, it is convenient to make measurements at low loads of <200g, the low load hardness is called microhardness [10,11]. There are three general types of hardness measurements depending on the manner in which the test is conducted. These are (i) scratch hardness (ii) indentation hardness and (iii) rebound or dynamic hardness. The Vicker’s hardness and different types of indentations are shown in Figs. 2.5 and 2.6.

A square-base diamond pyramid was used as an indenter in the Vicker’s hardness test. The angle between opposite faces of the pyramid is $136^\circ$. This angle is chosen because it approximates the most
desirable ratio of indentation diameter to ball diameter in the Brinell hardness test. Because of the shape of the indenter, this is frequently called the diamond –pyramid hardness (DPH) test. The DPH number or Vicker’s hardness (VHN), is defined as the load divided by the surface area of the indentation. In practice, this area is calculated from microscopic measurements of the length of the diagonals of the impressions. The VHN may be determined from the following equation

\[
VHN = \frac{2P\sin(\theta/2)}{L^2} = \frac{1.854P}{L^2}
\]  

(2.2)

where \(P\) = applied load in kg, \(L\) = average length of diagonals in mm and \(\theta\) is angle between opposite faces of diamond = 136°.

![Fig.2.5 Vicker’s pyramid diamond indenter indentation.](image-url)
2.9.5 Thermal Analyses

Thermal analyses involve a group of techniques such as thermogravimetric analysis (TGA), differential thermal analysis (DTA) and differential scanning calorimetry (DSC).

2.9.5.1 Thermogravimetric and Differential Thermal Analyses

Thermogravimetric (TG) curve can record the change in the weight of a sample as a function of temperature or time. Usually, the temperature range is from ambient to 1200°C with inert or reactive atmospheres. The change in weight may be due to dehydration or decomposition of the sample and therefore, a quantitative measurement of any change of weight associated with a transition is possible. The change in weight may also be due to the rupture or formation of various chemical bonds at elevated temperatures leading to the evolution of
volatile products or the formation of heavier reaction products. Thermogravimetric curve is characteristic of a given compound or system due to the unique change of physico-chemical reactions that occur over definite range of the temperature. The shape of thermogravimetric curve for a particular compound is influenced by the rate of heating of the sample and the surrounding atmosphere.

Differential thermal (DT) analysis was carried out for the samples to find the possible phase transition and to accurately determine the melting point. The behaviour of the compounds could be investigated systematically around the melting point from the DTA plot. A knowledge of the material decomposition is very important to explore the possibility of crystal growth from melt. In DTA the heating effects associated with chemical and physical changes of a substance are recorded when it is heated in a linear rate. In DTA, the temperature difference between the sample and a thermally inert reference material is continuously recorded as a function of furnace temperature or time. The thermal effects may either be exothermic or endothermic and are caused by physical changes such as fusion, crystalline structure inversion, boiling, vaporization, sublimation and others. Some enthalpic effects are also caused by chemical reactions such as dissociation or decomposition, oxidation, dehydration, reduction etc. Endothermic and exothermic peaks appearing on the thermogram will give information regarding
enthalpic changes. A transition undergone by the sample will result in the liberation/absorption of energy by the sample with a corresponding deviation of its temperature from that of the reference. The differential temperature $\Delta T$ vs the programmed temperature $T$ (at which the system had changed) enables the analyst to decide the temperature of transitions and also the nature of the transitions whether exothermic or endothermic.

The TGA and DTA analyses for the present samples were carried out from 30°C-700°C at a heating rate of 20°C/min in nitrogen atmosphere using a Seiko thermal analyzer available at Central Leather Research Institute, Chennai.

2.9.5.2 DSC Analyses

Differential scanning calorimetry (DSC) is also a thermo analytical technique in which the difference in the amount of heat required to increase the temperature of a sample and the reference are measured as a function of temperature. Both the sample and the reference are maintained at very nearly the same temperature throughout the experiment. The reference sample should have a well defined heat capacity over the range of temperatures to be scanned. The basic principle underlying the technique is that, when the sample undergoes a physical transformation such as phase transitions, more heat will be
needed to flow to it than the reference to maintain both at the same temperature. By observing the difference in heat flow between the sample and reference, differential scanning calorimeters are able to measure the amount of energy absorbed or released during such transitions.

A typical differential scanning calorimeter consists of two sealed pans: a sample pan and a reference pan (which is generally an empty sample pan). These pans are often covered by or composed of aluminium which acts as a radiation shield. The heat flux of DSC is shown in Fig. 2.7.

During the experiment, the instrument detects the differences in the heat flow between the sample and reference. This information is sent to an output device, most often a computer, and results in the plot of the differential heat flow between the reference and sample cell as a function of temperature. When there is no thermodynamic chemical process occurring the heat flow, the difference between the sample and reference varies only slightly with temperature, and shows up as a flat, or very shallow base line on the plot. However, an exothermic or endothermic process within the sample results in, a significant deviation in the difference between the two heat flows. The result is a peak in the DSC curve.
The sample is generally placed in an aluminum sample pan, which is then placed in the sample cell. The reference consists of a matched empty aluminum sample pan that is placed in the reference cell of the instrument. The sample pans are designed to have a very high thermal conductivity. Sample sizes generally range from 0.1 to 100 mg. The instrument cells are often airtight in order to shield the sample and reference from external thermal perturbations. This also allows experiments to be performed under variable pressures and atmospheres.

In the present study, the DSC and specific heat of the grown crystals are analyzed using a TA instruments (2920 Modulated DSC).

![Diagram of DSC setup]

Fig. 2.7 Heat flux of DSC.
2.9.6 UV-Vis Spectroscopy

Ultraviolet and Visible spectroscopy (electronic spectroscopy) is primarily used to measure the multiple bond or aromatic conjugation within the molecules. When a molecule absorbs light of a particular wavelength, an electron can be raised from its highest occupied molecular orbital to the lowest unoccupied molecular orbital causing an electronic transition. UV and visible light have sufficient energy to cause only the two electronic transition namely n-\(\pi^*\) and \(\pi-\pi^*\). This means that only compounds with \(\pi\)-electrons or nonbonding electron can produce UV-Vis spectra \([8,12]\). Hence, UV-Vis spectroscopy is very useful to measure the conjugated double bonds and also aromatic conjugation within the various molecules. It also distinguishes between conjugated and nonconjugated systems. Detection of chromophore in an unknown compound by comparison of its spectrum with that of a known compound is also possible. UV-Vis spectroscopy can be used to identify stable tautomeric species.

In the present investigations, UV-Vis spectroscopy is used to determine the optical transmittance window of the synthesized materials. Since NLO materials should have maximum transmittance in the UV as well as visible region of the electromagnetic radiation, optical transmittance spectrum is recorded for the synthesized materials using a Varian Cary 5E UV-Vis spectrophotometer in the region 200-800 nm.
2.9.7 Measurement of Refractive Index – Brewster Angle Method

The refractive index and its variation with temperature, i.e. the thermo-optic coefficient, are the basic optical properties of materials and are the backbone of the linear, nonlinear optical and opto-electronic devices. Several techniques have been used to measure the refractive index of the optical materials [13]. The angle of incidence at which the reflected beam is completely plane polarized is called the polarising angle. The tangent of the polarising angle is numerically equal to the refractive index of the medium. This is known as Brewster's law. In Fig. 2.8 the reflected and refracted rays are perpendicular to each other when a ray of light is incident at the polarizing angle on a refracting surface.

\[ n = \tan i_p \]  

(2.2)

Fig. 2.8 Brewster angle used for refractive index measurement.
2.9.8 Kurtz–Perry Technique-Powder SHG Efficiency Measurement

It is a difficult, time consuming, and costly endeavour to develop single crystal growth methods for new NLO materials. Therefore, a relatively rapid and simple method of screening new candidate materials for frequency conversion applications prior to crystal growth is needed. Kurtz and Perry [14] were the first to develop such a method. The advantage of their method is that it is inexpensive and not only provides a screening of some $d_{ij}$ values, but also determines whether a material is phase matchable or not. This has been proven to be a very useful method for screening new materials in powder form.

![Fig.2.9 Schematic diagram of Kurtz and Perry powder technique.](image)

The experimental set up for Kurtz and Perry powder technique used in the present investigation is shown in Fig.2.9. In this method,
the powdered sample is packed tightly in a capillary tube and illuminated using a Q-Switched Nd:YAG laser (1064 nm, 8 ns, 10 Hz). The laser power incident on the capillary tube is 6 mJ. The transmitted fundamental wave is absorbed by CuSO₄ (F1). F2 is a green filter, which will absorb residual fundamental wave (1064 nm) if any, transmitted by F1. ‘IF’ is an interference filter with bandwidth 4 nm and central wavelength of 4 nm. The SHG is confirmed by the emission of green radiation (λ = 532 nm). The amplitude of the SHG output voltage is measured using a 1 KV photomultiplier tube (PMT) and a digitalizing oscilloscope (CRO) assembly. In the present investigation, the SHG efficiencies have been measured with reference to the well known NLO material urea. The continuous increase of SHG intensity with the increase of particle size and remaining essentially constant at particle sizes greater than the average coherence length (Ic), confirms the phase matching behaviour of the material [15].

2.9.9 Laser Damage Threshold

It is known that the harmonic conversion efficiency is proportional to the power density of the fundamental beam. Hence, a convenient way to increase the efficiency is to focus the beam into the crystal. But it often leads to the breakdown of the materials, catastrophically damaging the crystal. It is then useful to prescribe the maximum
permissible power for a particular crystal, defined as damage threshold [16]. The surface of a material often tends to get damaged at power levels much lower than that for the bulk. Hence, this necessitates the independent definition of surface and bulk damage resistances. The minimum power level that causes damage to at least 50% of the sites irradiated is defined as the single shot (1-on-1) damage threshold ($P_1$). However, power levels much lower than this are detrimental to the material under continuous exposure. Hence, at $n$ pulses, damage may occur to the material at power levels ($P_n$) such that $P_n < P_1$. According to Nakatni et al. [17], the multiple shot (n-on-1) damage threshold is the minimum power level below which the crystal does not suffer damage even after 2000 pulses. However, the origin of laser damage threshold is highly complex [18]. It is largely material dependent. The morphology of the damage pattern often reflects the symmetry of the crystal surface. Earlier workers have predicted the possible origins of damage as thermal decomposition, photo ionization, ablation, photochemical dissociation, stimulated Brillouin scattering etc. [17]. The experimental setup used for this measurement is shown in Fig. 2.10.
2.9.10 Birefringence Measurement—Interference Technique

Crystals may be classified as either isotropic or anisotropic depending upon their optical behaviour and whether their crystallographic axes are equivalent or not. All isotropic crystals have equivalent axes that interact with light in a similar manner, regardless of the crystal orientation with respect to the incident light wave. Moreover, light entering an isotropic crystal is refracted at a constant angle and passes through the crystal at a single velocity without being polarized by interaction with the electronic components of the crystalline lattices.
Anisotropic crystals have crystallographically distinct axes and interact with light in a manner that is dependent upon the orientation of the crystalline lattice with respect to the incident light. When light enters the optical axis of anisotropic crystals, it acts in a manner similar to its interaction with isotropic crystals, passing through single velocity. However, when light enters a non-equivalent axis, it is refracted into two rays, each polarized so that they pass at different velocities and that their vibration directions are oriented at right angles to one another. This phenomenon is known as "double" or "bi" refraction and is seen to a greater or lesser degree in all anisotropic crystals. When double refraction occurs in an anisotropic crystal, light travels at two different velocities. One of the resulting polarized rays travels through the crystal with the same velocity in all directions and is termed as the ordinary ray. The other ray travels with a velocity that is dependent upon the propagation direction within the crystal. This light ray is termed as extraordinary ray. The distance of separation between the ordinary and extraordinary rays increases with crystal thickness [19].

Interference Technique

The Fig.2.11 shows the experimental setup of interference technique used for birefringence measurement [20]. It consists of a polarizer, analyzer, spectrometer and a halogen lamp. The polarizer
Fig. 2.11 Birefringence measurement – Interference technique.
and analyzer are placed in crossed positions and the crystal is placed
between them such that its optic axis is perpendicular to the incident
ray. A halogen lamp (500 W) is used as a source. The interference
pattern is observed through a high-resolution spectrometer.

Experimental Procedure

When the light enters the polarizer, it is polarized with an
orientation of the direction indicated by the arrow. Then the polarized
light enters the anisotropic crystal where it is refracted and divided into
two separate components (ordinary and extraordinary rays) that vibrate
parallel to the crystallographic axes and perpendicular to each other.
The polarized light wave then travels through the analyser, which
passes only those components of the light wave parallel to its
polarization direction. Interference occurs between the waves as they
pass through the analyzer. The net result of this interference will be
constructive (bright) or destructive (dark) lines. The interference pattern
is observed through the spectrometer. The wavelength of each
interference line is calculated from the calibration curve.

2.9.11 Construction of Electric Furnace

Any physical property of the materials depends upon the
temperature, pressure etc. To measure the birefringence as a function
of temperature a new electric furnace is designed and constructed.
(Fig.2.12). A kanthal wire of thickness 1.5 mm diameter is used as a heating coil. It is uniformly wound on a borosil glass tube of diameter 6 cm and the height 50 cm. Both ends of the kanthal wire are connected to the temperature controller through a relay with a dimmerstat. A Cr-Al thermocouple is placed on a heating coil and connected with the temperature controller. A Fe-K sensor is placed near to the sample holder to measure the exact temperature of the sample. The heating zone is surrounded by the glass wool to prevent the heat loss. A cylindrical metal of diameter 14 cm and height 55 cm covers the whole furnace and it acts as a radiation shield.

**Working**

By passing AC power through the furnace coil, heat will be generated. The temperature of the furnace can be changed using the temperature controller. On reaching the set temperature identified by the sensing thermocouple, the temperature controller switches off the power through the relay. The relay again switches on when the temperature decreases from the fixed temperature. The rate of heating of the furnace can be varied using the dimmerstat. The temperature controller controls the furnace temperature with an accuracy of ±1°C. The furnace can be operated up to 400°C.
Fig. 2.12 Electric furnace.
References


