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# Optical Techniques in the Determination of Pitch Lengths in the Cholesteric and Chiral Smectic C Phases

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## Abstract

There is no denying that the future of liquid crystals is in the display industry. The use of the twisted nematic (TN) and the superwisted nematic (STN) is widespread. The twisting in these devices is achieved either by use of optical components such as polarizers or the use of chiral dopants in a guest-host system. The later has gained much more attention. This is because not only are the accompanying optical components not needed, but any desired colour can be achieved by varying the pitch length of the helix formed by the chiral nematic (cholesteric) compound. In such application, the ability to determine the pitches of the resulting helical superstructure accurately and quickly is essential. Currently there are many methods for measuring the pitch of a helical super structure. These methods mostly employ optical or spectroscopic techniques. In this paper, four such methods namely, Fingerprint, Diffraction, Selective Reflection and Cano-wedge, are discussed in terms of their theoretical foundation, suitability to given situations and the validity of the results obtained. The suitability of the method depends among other things on the type of mesophase and the length of the pitch in relation to the wavelengths of the visible light. While the Fingerprint and Selective reflection may be used for both cholesteric and chiral smectic C (SmC*), Diffraction and Cano techniques are most suitable for the cholesterics.

**Keywords:** Chiral dopant • Cholesteric • SmC • Pitch length • Fingerprint • Diffraction • Selective reflection • Cano-wedge

## Introduction

It is an established fact that, the future of liquid crystals is in the display industry. Up to recently, of the many displays, the twisted nematic (TN) and the supertwisted nematic (STN) dominated this industry [1,2]. In both of these display types, the twisting may be done by use of either polarizer arrangements or the use of helical twisting power of chiral dopants in a guest-host system. At present the latter has gained more grounds, especially in the colour flexible displays. The reason for this is that, one can (at least theoretically), get any desired pitch length, thus any desired colour [3].

It has been known for quite some time that when a chiral compound is dissolved in a nematic liquid crystal or in the smectic phase, it induces its chirality into the phase [4-9] This is seen as a helical superstructure formed by the now chiral liquid crystalline phase. The chirality of the unseen solute molecules may be studied as it not only manifests itself in the helicity of the liquid crystal phase but also magnified several orders of magnitude [4-6,10].

The ability of the chiral molecules to twist as it were, the liquid crystalline matrix is what is referred to as the Helical Twisting Power (HTP) of the chiral molecule. This power of the chiral molecule in twisting the liquid crystal phase is normally determined from the amount of twist the phase has undergone. A ready measure of the twist is the periodicity of the helical superstructure formed by the phase. This periodicity is commonly referred to as the pitch of the helix. Hence the HTP of a chiral compound is defined as "the slope of the reciprocal of the pitch P, versus concentration C of the chiral molecules." HTP of the chiral molecule is thus given as [11,12].

$$\mathrm{HTP} = \frac{\lim 1/PC}{c \to 0} \quad (1)$$

The measurement of the HTP of a chiral compound is thus reduced to the determination of the helical pitch of the induced phase. This superstructure is shown in Figure 1.

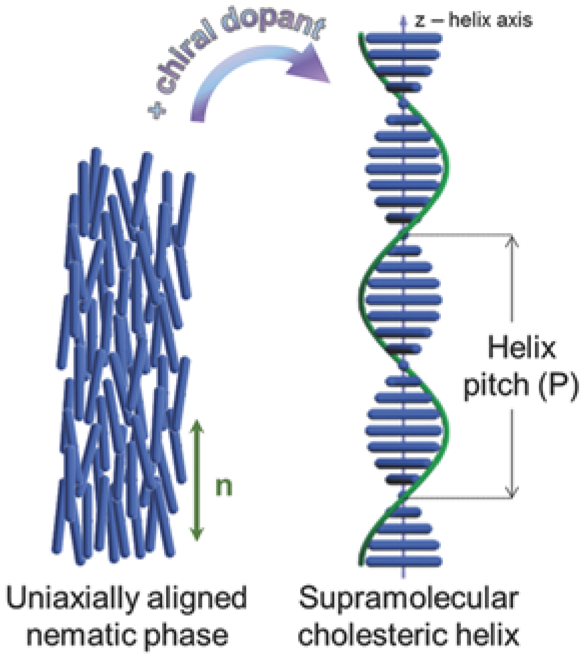


**Figure 1.** Formation of a cholesteric helical structure by doping nematic liquid crystals with chiral molecules, n is the director [13]

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Different methods for pitch measurement exist [13-17]. In this paper, four methods; the Grandjean-Cano, Fingerprint (Lagarde method), Diffraction method and Selective reflection are discussed. Recently there is much research interest in the use of cholesteric liquid crystals as diffraction gratings in optoelectronic devices [18,19].

These four methods which employ some form of optical techniques to measure the pitch of chiral compounds, will likely be more employed as research into the use of cholesteric liquid crystals, as diffraction grating gains momentum. Hence a review of their status is called for.

## Methods and Discussion

The preparation of samples for HTP measurements involves dissolution of the chiral dopants in an achiral liquid crystal. The concentration of the samples is so chosen such that there is enough of the dopant to twist the liquid crystalline phase, while making sure that all the dopant that is put in, completely dissolves.

The liquid crystal phase is always aligned so that the molecules are either parallel (homogeneous alignment) or perpendicular (homeotropic alignment) to the substrate. A good review of alignment procedures is described by Kuball et al. [20]. It should however be pointed out that, while the alignment of cholesteric phase is a fairly simple one, that of the smectic C (SmC) phase is not easy, as one needs not only align the molecules but also the smectic layers. This may at times require the use of electric or magnetic fields [21-23].

**Grandjean-cano method**

The Grandjean-Cano method of measuring the helical pitch is based on the observation that discontinuity lines appear when a chiral mesophase is put in a cell of variable thickness usually referred to as Cano wedge [24]. Such arrangement is easily obtained when two glass plates are set up to form a wedge with a small angle φ between them.

As you move upwards or downwards in the cholesteric or the SmC phase, the liquid crystal forms a helix, repeating itself after every period. For the cholesteric phase, this identity period comes about for every rotation of the director representing half the pitch of the helix. For the chiral SmC phase, the identity period comes about for every 2 rotation of the director, i.e. after every full pitch of the helix. Hence in the cholesteric phase the disclination curves separate regions which differ by half pitch, while for the chiral smectic C (SmC*) phase the regions differ by full pitch [25]. The case of the cholesteric phase is shown on Figure 2.

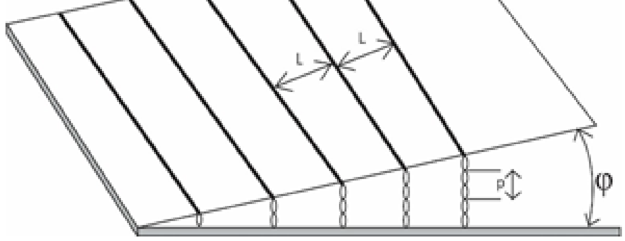


**Figure 2.** Formation of the stripped pattern (disclinations) by the cholesteric phase in a Cano wedge, when observed from above. φ Is the wedge angle, L is the fringes separation

From Figure 2, the line closest to the intersection of the two plates forming the wedge is formed by half pitch, the second line is one pitch, while the third line is for 3/2 pitches etc. the spacing between any two lines is therefore 1/2 p. The spacing L of the stripped pattern which appears parallel to the edge of the wedge, is related to the pitch of the helix (Figure 2) as per the equation; (p/2)/L=sinφ. where φ is the angle of the wedge, this simplifies to [26].

$p=2L\sin\varphi$ (2)

For small φ, p=2Lφ. The spacing L is measured directly, and pitch p is thus determined [26,27].

The Gradjean-Cano disclinations can only be observed in this Cano-Grandjean set up when the axis of the helix formed is perpendicular to the glass surface. For the cholesteric phase this comes about when the molecules are parallel to the glass surface, i.e., aligned homogeneously. As one moves up from the glass surface, the directors of molecules (which may be considered for this purpose to be in layer like structure) change direction as you move from one pseudo layer to another in a continuous shift, either to the left or to the right depending on whether the helix formed is right or left-handed respectively [28].

For the SmC* phase, the disclinations may only be observed when the molecules are aligned homeotropically. In such arrangement the SmC* layers are parallel to the glass surface and the helical axis is then perpendicular to the surface [29]. The tilt of the molecules precesses about the helix axis as you move from layer to layer. Again, the direction of precession whether clockwise or counterclockwise depends on the handedness of the helix.

To achieve the homogeneous alignment of the cholesteric molecules, the surfaces of both the lens and the glass plate are rubbed with Polyamide. For the homeotropic alignment of the SmC* molecules, the two surfaces may be rubbed with a solution of lecithin in diethylether [20]. The major problem with this simple Grandjean-Cano method has been its accuracy. The difficulty of measuring accurately the wedge angle φ, introduces large errors, in the range of 20% [26]. An improvement on the measurement of wedge φ, is the use of a planoconvex lens on a glass plate [27,30].

When a chiral phase is placed on a glass plate and a plano-convex lens is placed on it, under the experimental conditions described below, the Grandjean-Cano disclinations appear in the form of concentric circles. [14,26,31].

The Cano-Grandjean measurement basically involves observing the rings formed (as described above) under a polarizing microscope. With the aid of a camera directly above the sample the rings may be observed by simply looking into the eyepiece or better, on a video screen. The rings as in the case of the SmC* may not be clear, and rotation, (rather jerking) of the lens may help very much. The pitch of the helix obtained from the radii of the rings is determined automatically in some equipment or by direct measurement.

The pitch of the helical structure may easily be determined from the geometry of the arrangement as [26];

$p=((R_{i+1}^{2}-R_{i}^{2}))/r_{lens}$ (3)

Although by use of the plano-convex lens, the difficulty of measuring the small angle of the wedge was removed, a new problem, of aligning the curved surface of the lens is introduced. Besides, this method is still found to be cumbersome [26].

Podolskyy et al [26] report an improvement on the earlier two plate wedge method, using what they called “stripe-wedge” method to measure the pitch.

Generally, this is a very accurate method for pitch lengths much longer than the wavelengths of visible light [27]. However, it is recommended mostly for the cholesterics as the pitch in SmC* are much longer than the wavelengths of the visible light. The limit to the use of this method comes about when p is comparable or shorter than the wavelengths of visible light, as then φ must be made very small, which is difficult [27] Another reason why this method is not recommended for Smectics is because the Smectics are viscous and the rings are very irregular and are thus not very suitable for pitch measurement [27].

**Fingerprint (Lagarde method) method**

This method essentially consists of looking at a homeotropically aligned sample between two microscope slides (sample cell), under a polarising microscope. Under such alignment of the cholesteric phase, the axis of the helix lies in the plane of the liquid crystal cell. The period of the helix is determined by its pitch. [19] When the sample is illuminated using monochromatic light, some equal distant dark lines (finger like fringes) are formed perpendicular to the helix axis, the spacing between which yields half the pitch length as explained below [32].

The origin of the fringes and their separation has been a subject of much discussion [21,22,33]. It is now an established fact that the observed fringes are the result of a number of factors including wave surface distortion, optical neutral lines or ray deflection, or even a mixture of some of these [33]. Also, the cause differs between the cholesteric and the SmC* phases.

The crest-like and trough-like features on the helical superstructure have different refractive indices, causing variations in the velocities of light passing through them. Hence creating wave like distortions on the surface. This is what is usually referred to as wave surface distortions [33]. For small distortions, the surface acts as a thin lens. The troughs (concave regions) of these surface waves converg

e the rays reaching them, forming real images above the sample, while the crests or the convex regions, and diverge the rays, forming virtual images below the sample. These images consist of equal distant lines.

Focussing the objective above or below the sample yields fringes whose separation is half the pitch. Remember they are a result of light from either crest (or troughs) which are always one half the wavelengths. Focussing on the middle of the sample yields an overlap of the two sets of fringes where lines from one goes in between the others, leading to a spacing of quarter pitch. These types of fringes are usually not very clear, as the objective is not essentially focussing on them [32].

By varying the cell thickness, and the elastic constants anchoring coefficients, among other parameters, and with the homeotropic alignment, highly uniform lines with regular patterns can be obtained [18]. From which the pitch may be determined.

Though this method is a simple one, the results are sometimes contentious due to the confusion regarding as to which line spacing represents the pitch. Also, these variations in the spacing in the same sample often yields values of pitch, which are not in agreement with other methods [32].

Dietrich et al [32] point out that this being a visual technique; only pitch lengths longer than 1.5 μm can be observed easily. However, by use of a polarized laser beam, much smaller pitch lengths can be measured [32].

**Diffraction method**

When the sample orientation is homogeneous, so that the helix axis is parallel to the substrate surface, the texture acts as a many-slit diffraction grating, whose spacing (or grating constant) gives a direct measure of the pitch [32-35]. The usual grating equation in equation 4 applies.

$$d \sin\theta = m\lambda \quad (4)$$

Where  is the diffraction angle of the mth order image with respect to the central fringe, λ is the wavelength of the light used, d the usual grating element represents the pitch.

A sample with planar orientation is prepared in the same way as in the Finger-Print method and observed under a polarising microscope and the most oriented finger-print texture is chosen. This aligned texture is then placed in the path of a He-Ne laser beam in a spectrometer. The diffraction pattern so obtained is analysed in the usual way and the grating constant determined [32,36]. Care must be taken, as the laser is dangerous to the eye.

From the angle corresponding to the different orders of diffraction, the pitch, represented by the grating spacing (also known as grating element), may thus be easily determined. The method is suitable for long pitch lengths in relation to the wavelengths of the visible light [26].

Though this method is an accurate one, its application is however restricted to the situation where the orientation of the helix is homogenous. Since homogenous alignment of the smectic phase is fairly difficult due to the layer structure of the phase, this method is commonly used for the cholesteric phase.

The alignment of Smectic liquid crystals is not easy generally. According to Ingo Dierking [27], what is known to work perfectly well with one compound may not work at all with a similar compound. However, in general to obtain a planar arrangement he advises the use of polymer films deposited on glass substrates. Homeotropic alignment maybe achieved by depositing surfactants on glass substrates [27].

**Selective reflection method**

In the selective reflection method of determining the pitch of a chiral compound, the sample is placed between two microscope slides whose surfaces were treated such that, the helical structure of the phase is uniformly aligned, and the helix axis is normal to the substrate surface [30]. The sample may be placed between two glass prisms or between a glass plate and a prism [37]. The sample is then transferred to a spectrophotometer and its transmission spectra determined as it is cooled from the isotropic phase to the required liquid crystalline phase. The transmission spectra may be determined at either normal incidence or oblique incidence [25,27,38].

The transmission spectra usually consist of one peak at the normal incidence (for cholesterics) or two peaks for the normal and oblique incidences respectively in the case of the $SmC^*$ phase. The second band in the case of oblique incidence occurs at a wavelength approximately twice the first peak of the normal incidence [39].

When a narrow wavelength range about the wavelength ( ) of the incident light beam is directed parallel to the helical axis of the superstructure, the beam is split into its left and right circularly polarised components by the helical structure [30]. This is the idea of optical activity. The component that has the same rotation sense as that of the screw direction of the helix is reflected, while the other is transmitted. Thus, circular dichroism takes place.

$\lambda = np$ (5)

Where p is the pitch of the helix and n is the mean refractive index, $n=(n_e+n_o)/2$ within a plane normal to the helical axis and is the wavelength of the incident light. ne and no are the extraordinary and ordinary refractive indices [26,40].

Now $\Delta\lambda=\Delta np$, where $\Delta\lambda$ is the bandwidth, $\Delta n$ is the birefringence ($n_e$-$n_o$) of the liquid crystal, and p is the pitch as above.

If the birefringence $\Delta n=(n_e-n_o)$ is measured and $\Delta\lambda$ is known, then the pitch can be determined. [26,40]. Moreover, as the peak position is found to vary with the angle of incidence, more values of the pitch may be obtained, and the mean found [39].

The intensity of selectively reflected monochromatic light depends on a number of factors, among them, the impurity and the composition of the sample as well as the degree of alignment of the sample. While impurity in the sample can cause a shift in the position of colour bands, poor alignment of the sample may lead to a mixture of domains, and the individual domains may exhibit different colours [40]. Hence an improvement in the quality of results from a selective reflection method, viz a viz the accuracy of the measured pitch, depends on the purity of sample and its degree of alignment. Improvement in the two leads to improvement in the accuracy of the measured pitch.

This method may be used for both the cholesteric and SmC* phases. One important condition for the use of this method is that the pitch p should be of similar magnitude as the wavelength of the light used or shorter [35].

Since the pitch in the SmC* phase is usually too long for wavelengths in the visible range, selective reflection cannot work in SmC phase but is mostly observed in the cholesteric phase [26,37]. For the very long pitches (much longer than the wavelengths in the visible light), the Cano-wedge technique described above comes in handy [37]. When the films are relatively thin, no clear selective reflection can be observed.

**Limitations of study**

Although there are many methods for determining the pitch of a chiral compound, in this review we looked at only four commonly used ones. These methods which employ optical techniques are chosen for this review because they may soon attract more attention as research into the use of cholesteric liquid crystals as small diffraction gratings, in optoelectronic devices gains ground.

## Conclusions

There are a number of methods in use for the determination of the pitch length. When using these methods their suitability to given situations and possible shortcomings need to be taken into account.

For long pitches in relation to the wavelengths of the visible light, Cano-wedge, Diffraction, and Fingerprint methods are best suited.

For pitch lengths in the range of the wavelengths of visible light, the Selective Reflection method is recommended.

Besides the confusion concerning which line spacing represents the pitch, Fingerprint method may be used for both cholesteric and smectic. Selective reflection may also be used for both.

Since the homogenous alignment is fairly difficult the diffraction technique is commonly used in the cholesteric phase.

Finally, due to the viscous nature of the chiral smectic C phase, the Cano method is recommended for cholesteric phase only.

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