CHAPTER 3

POLARIZATION MICROSCOPY

3.1 THEORETICAL ASPECTS

Polarization microscopy is by far the most powerful technique for the examination of textile fibres. Just a few elementary operations are needed to obtain a wealth of information on the sample. Polarization microscopy has its own specialized terminology. Before discussing the application of polarized light for man-made and natural fibres, it is inevitable to look at some basic but essential theoretical concepts.

3.1.1 Light and its polarization states

Light is electromagnetic radiation in which the components of the electrical field (E) and the magnetic field (H) are transverse waves that lie in planes perpendicular to each other. Light moves in a rectilinear way on the intersection of these planes, as shown in **[Fig. 3-1]**. The maximum speed of light approximately is $c = 3 \times 10^8$ m.s^{-1}.

There is a simple relationship between the speed of light in vacuum c, the wavelength λ and the frequency ν.

Speed of light $\mathbf{c = \lambda \, . \, \nu}$

The human eye is sensitive to radiation with wavelengths between 380 and 780 nm. This wavelength range is called visible light. The energy of light and its specific frequency are proportional; they differ by a constant factor equal to Planck's constant:

Energy of light $\mathbf{E = h.\, \nu}$

3.1.7 Orientation

The orientation is the alignment of polymer chains according to the fibre axis in both crystalline and amorphous regions. The orientation is higher if more chains are positioned parallel to the draw direction or, in other words, if fewer polymer chains deviate from the fibre axis.

The total orientation can be determined using the birefringence, which is the difference between the refractive indices in the two main fibre directions. The crystalline orientation is determined using X-ray analysis. The amorphous orientation can be derived from the degree of crystallinity and the crystalline orientation. The total orientation lies between 0 (randomly oriented polymer chains) and 1 (perfectly aligned polymer chains).

An increase in crystallinity does not necessarily mean an increase in orientation. This is illustrated in **[Fig. 3-6]**. Polymers can possess a high crystallinity but have a low orientation. Also, a polymer with low crystallinity can have a high degree of orientation.

The final crystallinity and orientation of the fibre is influenced by

- the properties of the raw polymer (chemical structure, molecular weight);
- the production process (polymerisation, spinning, drawing, texturizing, heat setting); and
- the dyeing process.

During each phase in the production process, different conditions are present, which affect both crystallinity and orientation. The most important parameters are temperature, tension (drawing force) and time.

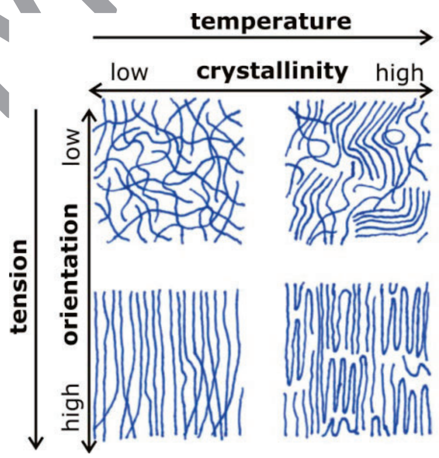


Fig. 3-6 Fibre crystallinity and orientation.

the right or lower border of the diagram. The Michel-Lévy colour chart was first developed for mineralogy and is still of importance to determine minerals. Pure minerals have a specific birefringence. Therefore, minerals are indicated on most charts, along with their corresponding birefringence values. Polymers often exhibit a range of birefringence values. The only textile fibres in the chart are silk (0,055) and nylon (0,060).

In the upper border of the diagram, the sample thickness is represented. One can easily see that materials with varying sample thickness, such as fibres, show a variety of different polarization colours. For fibres having very low birefringence, the lines of equal birefringence are positioned almost horizontally. As the birefringence increases, the "iso-birefringence lines" become increasingly steeper, which indicates a more significant change in interference colours.

The first order represents the colours of low birefringence. It sets off for the first 250 nm with black, grey and white. From thereon, yellow, brown-yellow or orange colours follow until first-order red at 530 nm is reached. The first order ends at about 550 nm with the first-order violet.

Bright colours characterize the second order. It starts with violet, then continues with second-order blue, green, yellow, orange, red and ends with second-order violet at 1100 nm.

The sequence of these colours is repeated in the third order, but the colours are becoming paler. The third order ends at 1650 nm.

Only the first three orders are entirely reproduced on the colour chart. At higher orders, the colours become increasingly faint until, eventually, high order white is achieved. It makes no sense to represent these colours because one would not be able to differentiate the colours.

The Michel-Lévy colour chart is beneficial for fibre examinations. It can be used to check the order of the interference colours and obtain an estimation of the birefringence. Also, using the colour chart, information on the fibre section can be obtained. These aspects are explained further on.

Fig. 3-13 Lambda plate or first-order red plate.
The two possible orientations of the turnable lambda plate corresponding to the slow direction are shown.

3.2.5 Quartz wedge

Although it is not essential, a quartz wedge can be used to check the order of the interference colours in a fibre. This tool has a wedge-like shape with increasing thickness. When the quartz wedge is inserted gradually deeper in the compensator slot, the increasing optical path provokes larger retardation values up to several orders. Subtracting a certain amount of orders allows reaching compensation. Using this procedure, one can determine the order without exactly measuring the birefringence. The number of orders of this **variable compensator** and its slow direction is indicated on the quartz wedge holder, as shown in **[Fig. 3-14]**.

Fig. 3-14 Quartz wedge.

The effect of a lambda plate on a low birefringent fibre with positive elongation is illustrated in **[Fig. 3-19]**. The fibre showing a grey interference colour under crossed polars turns blue if a lambda plate is placed in a parallel position. The fibre turns red if a lambda plate is inserted in a perpendicular position.

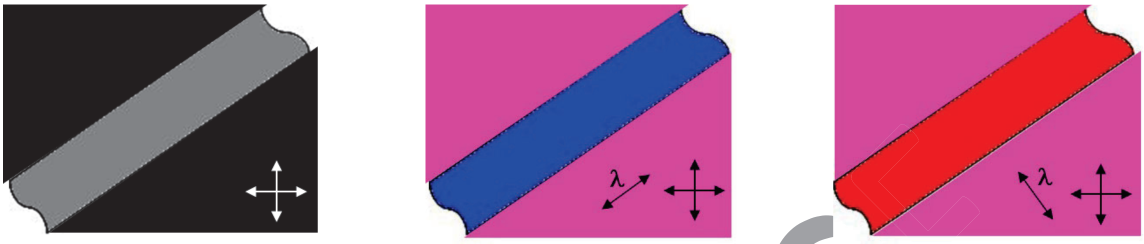


Fig. 3-19 The effect of a lambda plate on a low birefringent fibre with positive elongation.

The parallel position, i.e. lambda plate and fibre are aligned, is an **addition position**, i.e. the slow directions of both fibre and lambda plate correspond. The perpendicular position, i.e. fibre and lambda plate are orthogonal, is a so-called **subtraction position** for which the slow directions are in opposition.

Suppose a situation where the fibre has a slightly negative birefringence ($n_{\perp} > n_{//}$). When adding one order (550 nm), the interference colour of the fibre shifts from grey to the first-order red zone (retardation about 450 to 500 nm), as shown in **[Fig. 3-20]**. If one subtracted one whole order, a second-order blue (600 - 650 nm) would result.

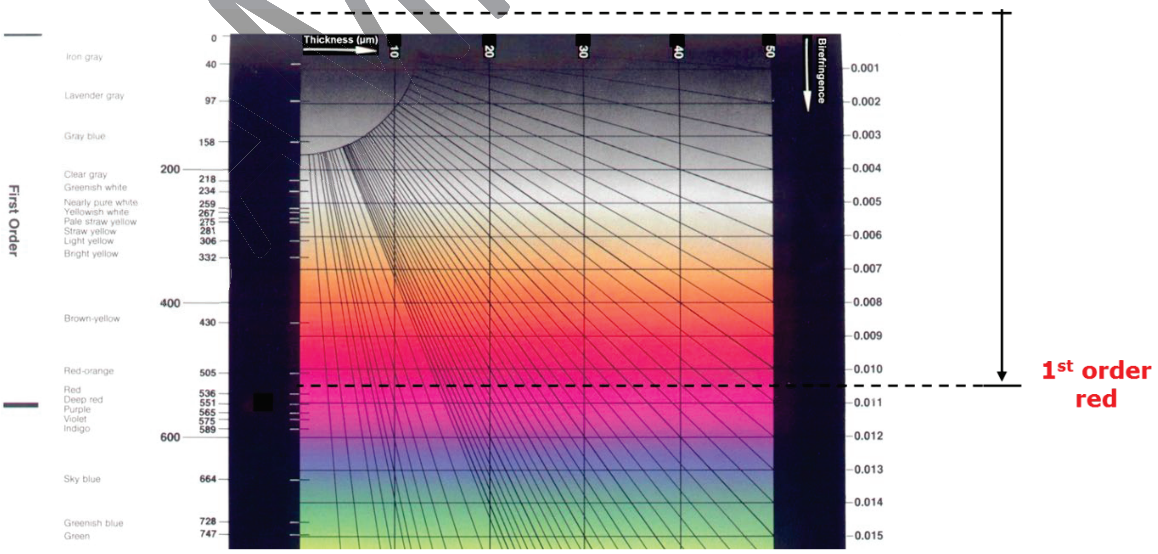
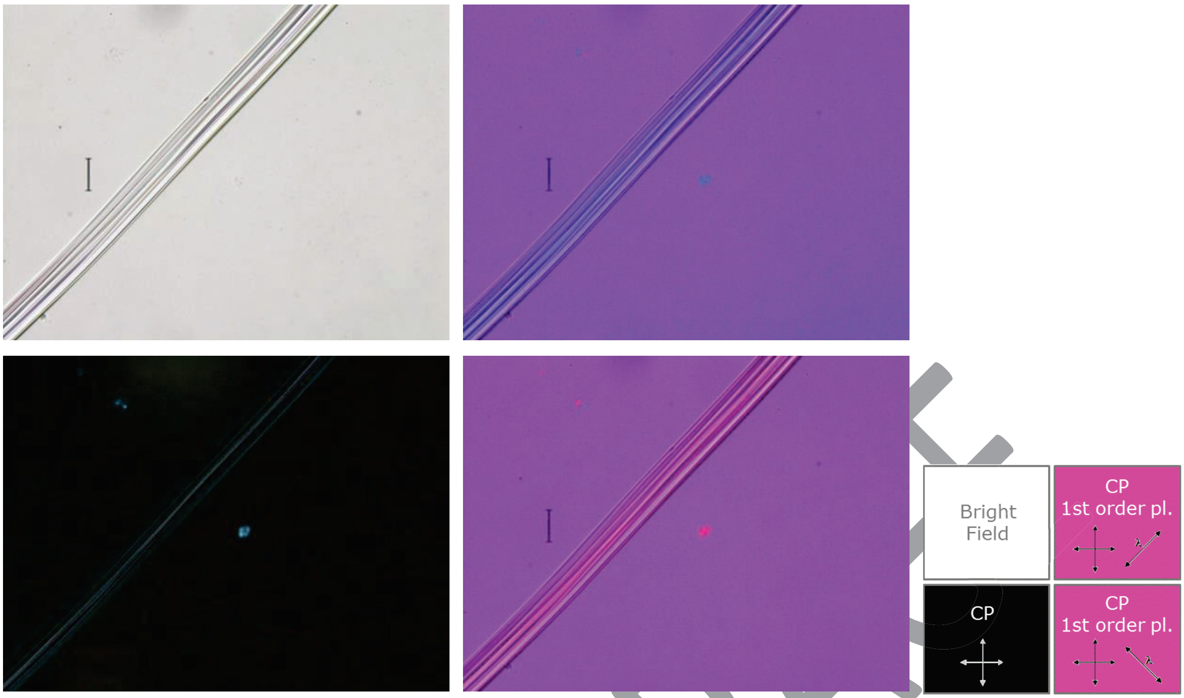


Fig. 3-20 Addition of one order for low birefringent fibres with negative elongation.



Ph. 3-5 Triacetate fibre viewed in bright field and with polarized light.

Triacetate is an almost isotropic fibre with birefringence values ranging from -0.001 to 0.001. The first order red and blue interference colours are very faint. This is illustrated in **[Ph. 3-5]**.

3.4.2 Low birefringent fibres

Fibres with low birefringence produce first-order colours. A good example is the viscose fibre, shown in **[Fig. 3-24]**. These fibres have an irregular fibre section, and for standard viscose, the thickness usually lies between 15 and 35 µm. Between crossed polars, this type possesses a first order orange-red, i.e. a brownish band (retardation about 500 nm) that does not necessarily lie in the centre of the fibre. Adding one order causes this band to shift to a second-order bright orange-red (retardation around 1000 - 1100 nm). The fibre edges shift from first-order white to a first-order blue in the addition position. Subtracting one order causes the brownish band to shift to near black (almost complete extinction) with surrounding first-order grey colours. The fibre edges are first-order red in the subtraction position.

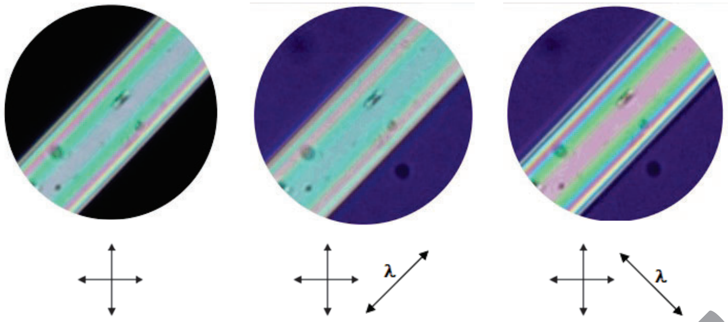
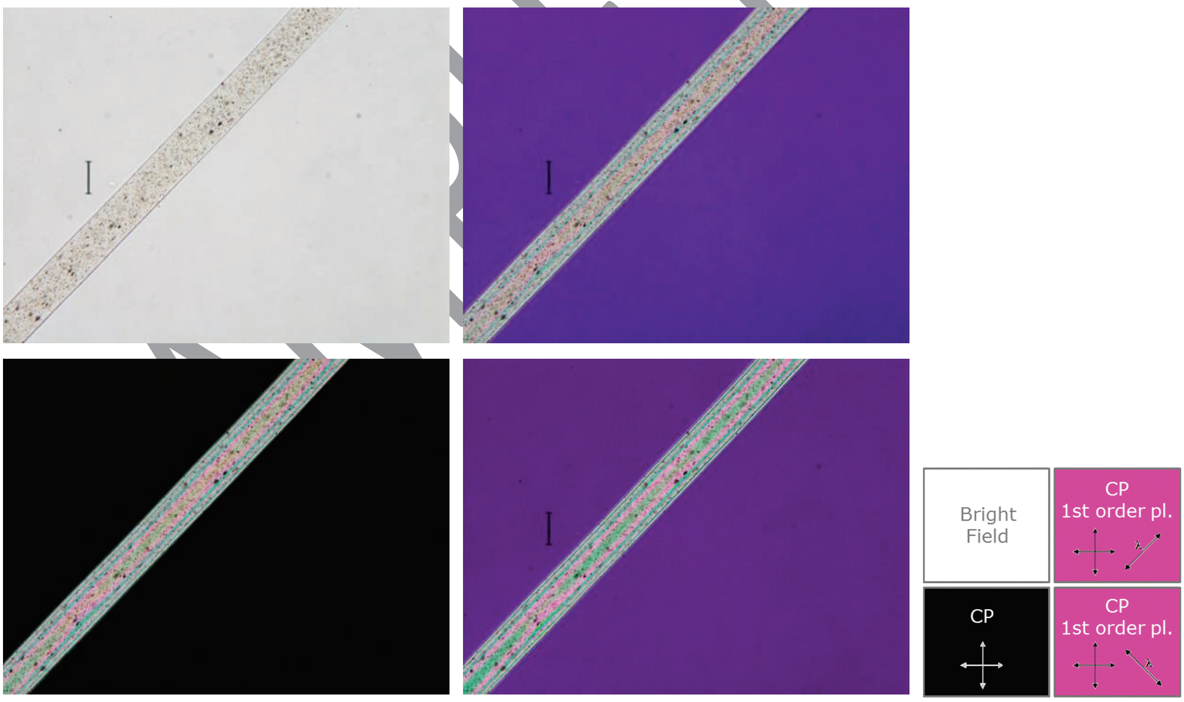
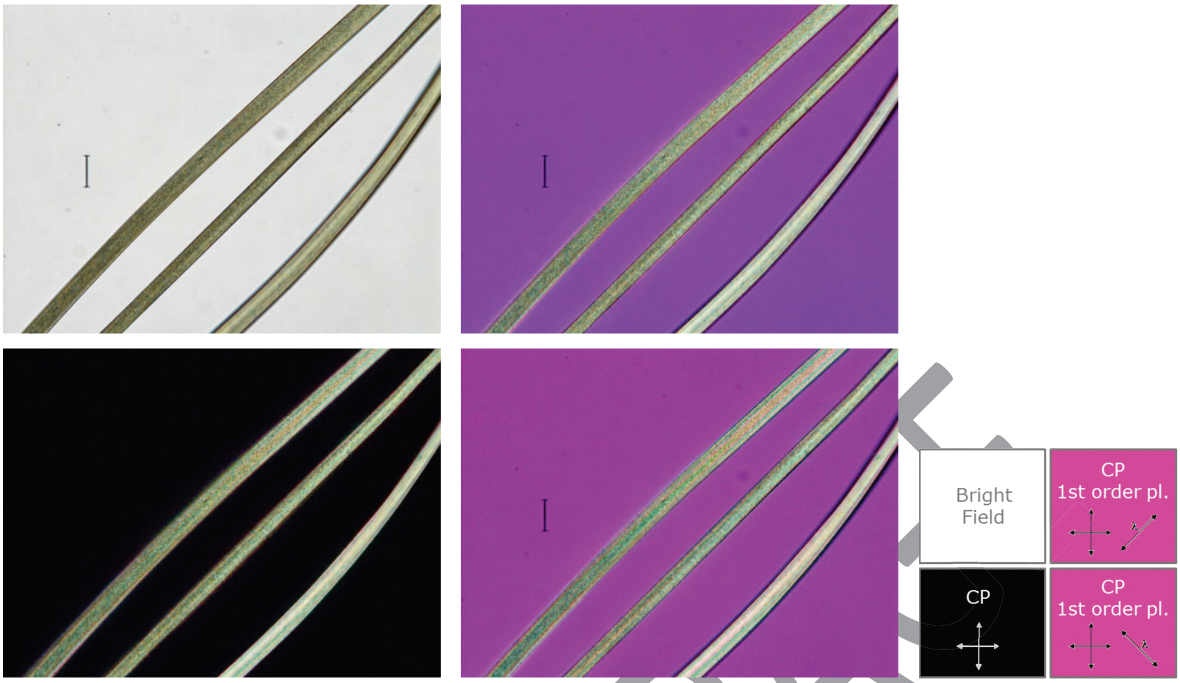


Fig. 3-27 Polarization colours of polyester (PET).

The PET fibre in **[Fig. 3-27]** has a circular section and a diameter of 12,5 µm. Under crossed polars, it presents a central faint pink colour. The retardation of about 2200 nm corresponds with the transition between the fourth and the fifth order. Adding one order produces very faint polarization colours; subtracting one order produces a violet-grey central band. This corresponds with retardation of about 1650 nm, which is the transition between the third and the fourth order.



Ph. 3-9 Polyester (PET) fibre viewed in bright field and with polarized light.



Ph. 3-14 A polyamide-imide (Kermel) viewed in bright field and with polarized light.

3.4.6 Polarization colours and cross-sections

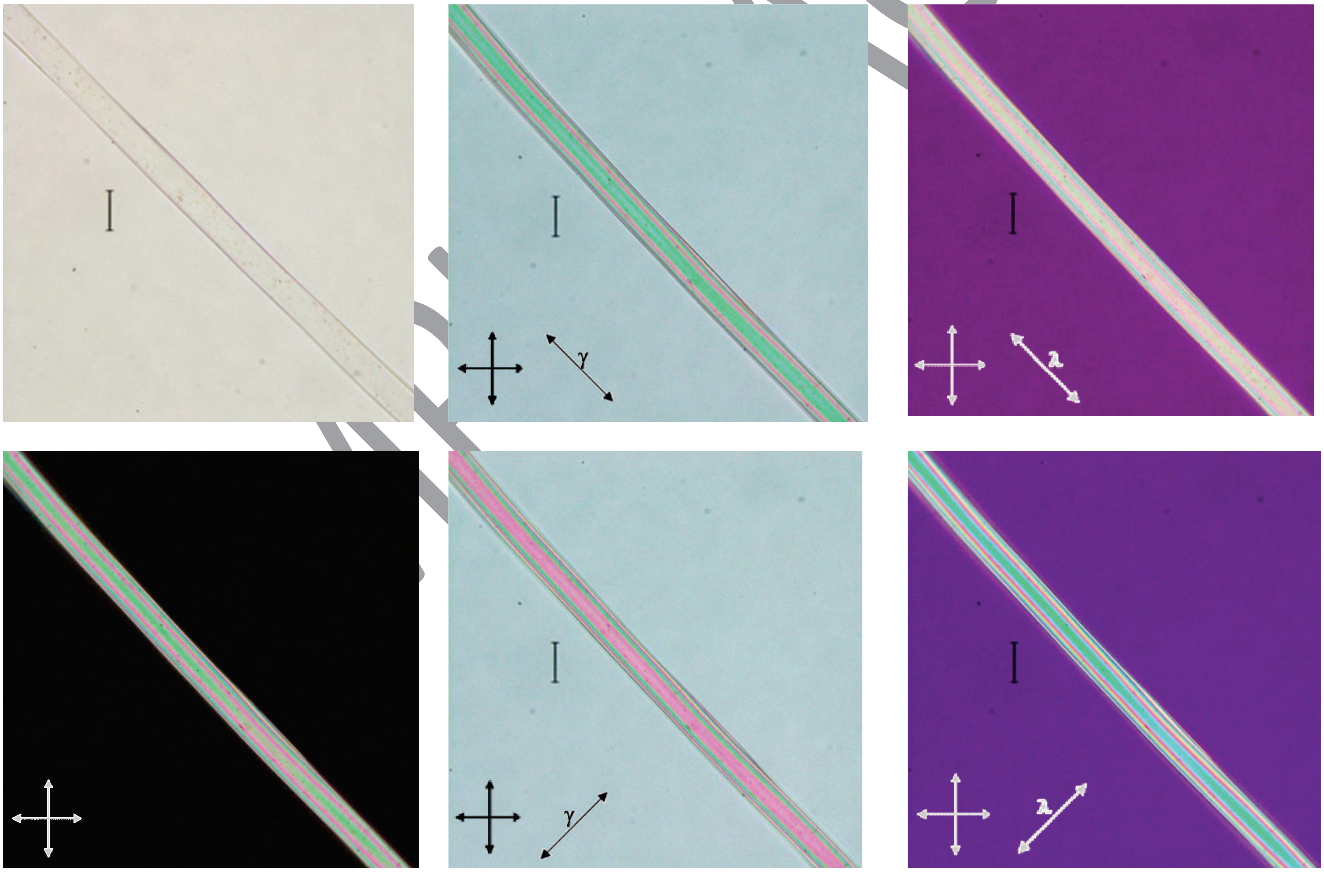
The cross-sectional shape of fibres causes a gradation in sample thickness. In zones of similar optical path, matching polarization colours appear in bands. Therefore, observation of the polarization colours, their distribution within the fibre and the band symmetry help determine the fibre section.

If a fibre has a perfectly **circular cross-section**, a symmetrical disposition of coloured bands around a broad central band is observed. The differently coloured bands next to the central band decrease in width towards the borders. These colours correspond to smaller retardations in the Michel-Lévy colour chart. Different colours are related to different path lengths. Hence, the succession of those different colours follows the curvature of the fibre. For circular sections, the maximal thickness corresponds with the diameter. At the borders, the thickness equals zero. Looking at the Michel-Lévy chart, one can follow the line of equal birefringence, from the maximal thickness towards ever-decreasing thickness. All polarization colours that lie on that line are present in the fibre. However, at both edges of the fibre, the curvature is very steep, and the different colours succeed each other very rapidly, i.e. the central band is relatively large. In contrast, the coloured bands become very thin towards the edges.

The polarization colours reflect the optical path length, and zones of equal colour represent equal thickness. The polarization colours can therefore be interpreted as a topographic map with lines of equal height. Different fibre types of polyethylene terephthalate (PET) are shown in **[Ph 3-20]**, each with different cross-sections. Circular and triangular cross-sections produce the most regular interference patterns.

3.4.7 Estimating birefringence using the Michel-Lévy colour chart

The colour chart can be used to estimate the birefringence. For fibres with a circular cross-sectional shape, the diameter of the fibre corresponds to the optical path. For all other sections, correction factors can be used to calculate the optical path. The central interference colour has to be located on the colour chart.



Ph. 3-21 The use of a fixed compensator.
Above: polyester fibre in bright field; CP + 1/4 λ; CP + 1 λ (addition position);
below: polyester fibre observed between crossed polars (CP); CP - 1/4 λ; CP -1 λ (subtraction position)
Magnification 630x (I = 16 µm)

Use can be made of a fixed compensator such as the lambda plate or a quarter-wave plate (λ/4) to locate the central polarization colour on the chart by subtracting (or

3.4.8 Quantitative polarization microscopy

It is not the aim of this work to describe quantitative polarization microscopy in detail. However, this topic is discussed here as it can help in gaining insight into polarization microscopy. Also, we did not find a clear description of the determination of birefringence in the forensic literature.

The best method to determine birefringence is by using a tilting compensator. This device consists of a calcite or quartz plate of a fixed thickness. This crystal can be tilted under a certain angle to obtain compensation. Each tilting position corresponds to a different optical path and retardation of the compensator crystal. The compensator has been calibrated, i.e. the tilting angles of the compensator are correlated with retardation. At compensation, the precise and known amount of retardation that was subtracted corresponds to the central retardation of the fibre. Therefore, high precision birefringence measurements can be performed with the tilting compensator. Several types of tilting compensators are available. The most useful compensators for fibre work are the Berek (or B) compensator and the K-compensator, shown in **[Fig. 3-28]**. Which of these compensators to use depends on the retardation range needed to obtain compensation. Low to medium birefringence fibres can be measured with the Berek-compensator (up to 4 orders). High birefringent fibres are usually measured with the K-compensator, which allows the determination of retardations up to 30 orders.

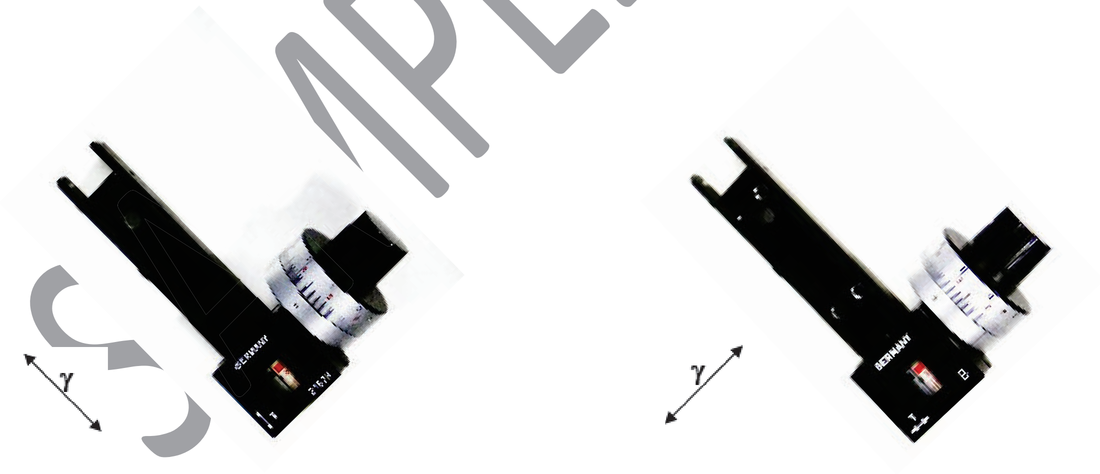


Fig. 3-28 Two types of tilting compensators.
The slow directions are indicated.

The birefringent crystal of the tilting compensator has a direction for which the refractive index is highest. This slow direction is usually indicated on the compensator with the Greek letter gamma γ.

A correction factor should be used for other cross-sections. In this case, the error in the determination of the birefringence is more significant.

☞ *The measurement of birefringence is mainly used to compare the polymer orientation in non-dyed fibre traces with reference fibres. The method is only recommended for fibres with a circular cross-section. Birefringence measurements cannot replace infrared or Raman spectroscopy to identify the generic fibre class and subclass.*

A tilting compensator is used for measuring fibre birefringence. This is a variable retarder; it has a birefringent crystal that can be inclined to increase the retardation that is subtracted from the retardation of the fibre. This compensator is calibrated, i.e. each inclination angle corresponds to a certain amount of retardation.

Compensation can only be obtained if the slow directions of both compensator crystal and fibre are brought in opposition (also called subtraction position). Compensation occurs when the retardation at the fibre centre is compensated by the retardation of the compensator crystal. This produces a total extinction of all wavelengths.

3.4.9 Observation of dichroism

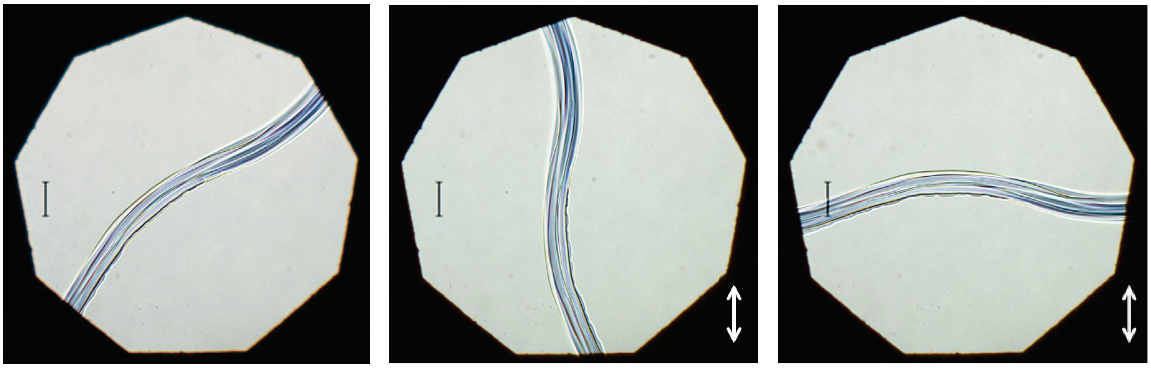
Dichroism is the selective absorption of polarized light of different orientation, corresponding to the two main fibre directions. Dichroic fibres display a change in intensity, a change in hue or both when observed with plane polarized light of different orientation. As linear polarized light is used, this is called **linear dichroism**.

The observation of dichroism requires the use of only one polar at any time. Two different observation methods are described here.

If the microscope is equipped with a circular stage, it is easy to use a north-south oriented polar and turn the stage over 90°, as shown in **[Fig. 3-31]**. The two main observation positions are:

- parallel position: north-south orientation of both fibre and polarization direction
- perpendicular position: east-west fibre orientation, north-south polar orientation

If the fibre is dichroic, a gradual intensity or hue change is observed when turning the stage from the parallel position to the perpendicular position.



Ph. 3-34 Weak regular dichroism in a diacetate fibre.
Left: bright field, middle: parallel position; right: perpendicular position.

The observation of linear dichroism is a fast and straightforward method. It is highly recommended to check the dichroic effect systematically in casework and use it as an additional characteristic to compare fibre traces and control fibres.

☞

Linear dichroism is the selective absorption of linear or plane polarized light by one of the main directions of a dyed or pigmented fibre. It is a measure of the alignment between dyestuffs and polymer chains.
The dichroic effect depends on:

- *the nature of the dyes or pigments;*
- *the nature of the fibre;*
- *the orientation of the polymer chains (birefringence).*

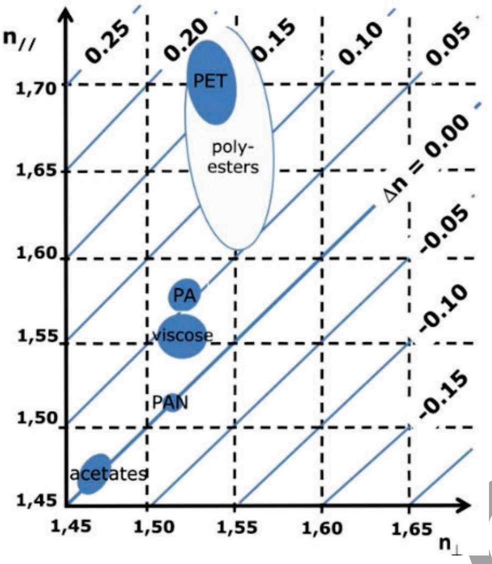
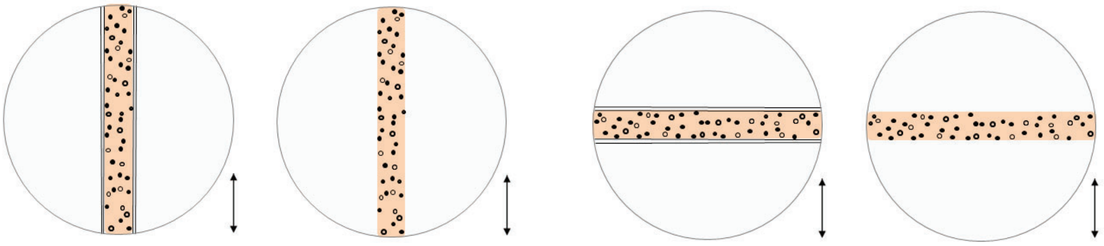


Fig. 3-35 Refractive index chart.
The variation of both refractive indices in the main man-made fibre classes is shown.

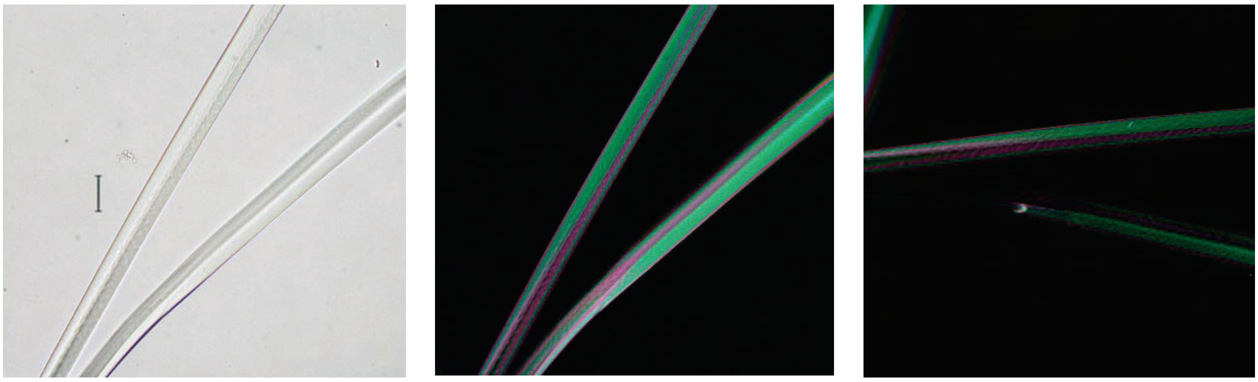
3.4.11 Dispersion staining technique

In the previous section, the DB displacement was used to verify whether the fibre or the mounting medium has the highest refractive index. If only one polar (N-S orientation) is used and the fibre is oriented N-S or E-W, this activates one of the refractive indices, $n_{//}$ or $n_{\perp}$. If the activated fibre refractive index differs a lot from that of the mounting medium (n_M), strong contrasting DB lines are observed. The DB lines shift quickly if the stage is lowered. If the refractive indices lie close together, the contrast is less and the displacement slower. If the refractive indices of fibre and mounting medium are equal, no DB lines are visible. In this case, the light can travel freely from the mounting medium to the fibre and vice versa without being refracted, as illustrated in **[Fig. 3-36]**.



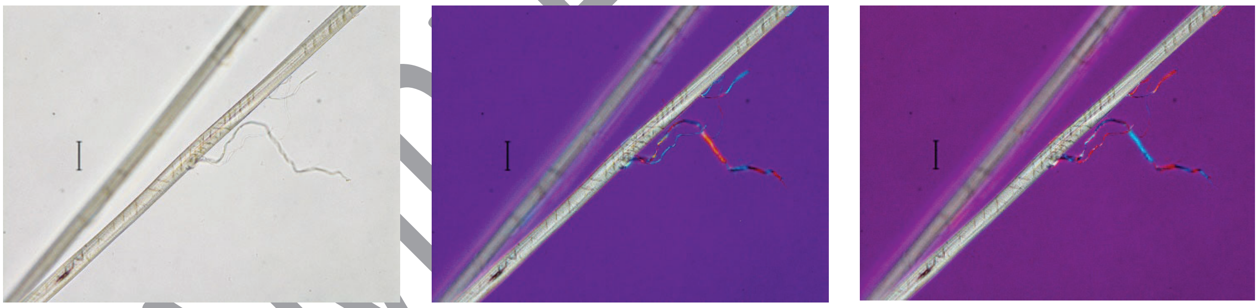
$n_{//} \neq n_M$ $n_{//} = n_{M'}$ $n_{\perp} \neq n_M$ $n_{\perp} = n_{M''}$

Fig. 3-36 Dispersion staining technique.



Ph. 3-43 Characteristic pattern of Nomex between crossed polars.

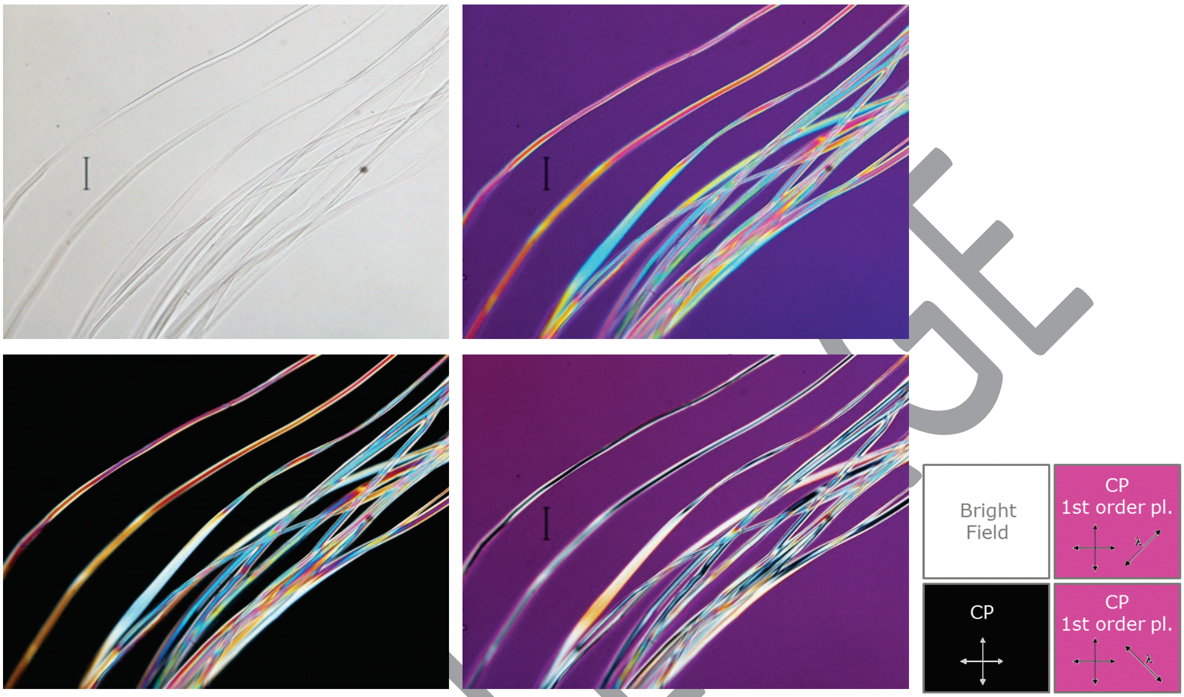
If a lambda plate is used in combination with crossed polars, other characteristics can be verified. The Kevlar fibre in **[Ph. 3-44]** shows **fibrillation**. Even the finest fibrillation can be detected when using crossed polars and a lambda plate. Because of its fineness and winding course, fibril sections are alternating blue (for addition positions) and red (for subtraction positions).



Ph. 3-44 Fibrillation in Kevlar fibres.
Fibres viewed in bright field (left), between crossed polars and lambda plate added (centre) and with lambda plate subtracted (right).

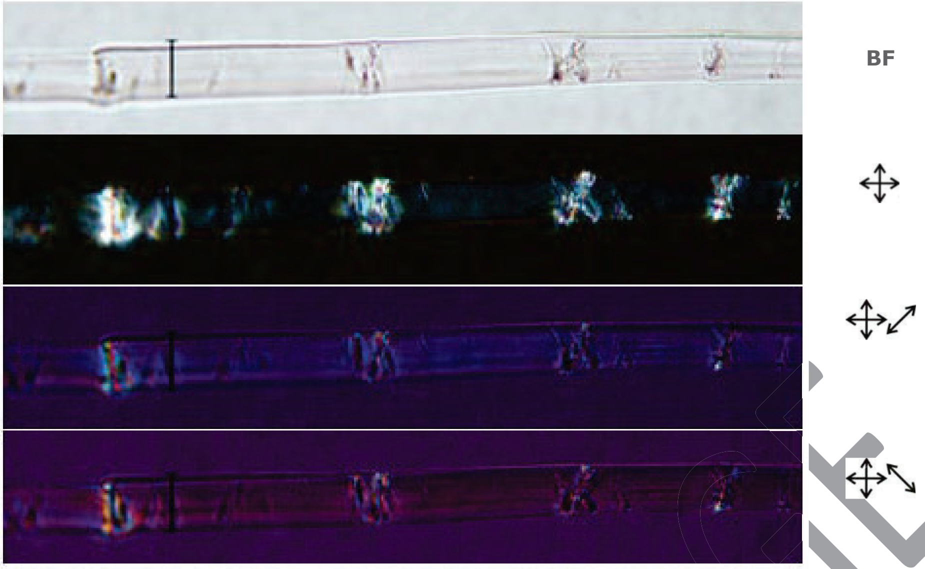
Likewise, due to their small diameter, **microfibres** do not show any polarization colours between crossed polars. The elongation of the microfibre tuft in **[Ph. 3-45]** was verified using crossed polars and a lambda plate. The whole microfibre tuft turns blue when the lambda plate is oriented along with the microfibre filaments. This indicates a positive elongation and a fibre composition that is most likely polyester or polyamide.

In mercerized cotton, the fibre has swollen to a more tubular form. Between crossed polars, the polarization colours are bright and more regular, as shown in **[Ph. 3-50]**. Subtracting an order can cause extinction for some fibre regions, as is the case for viscose.



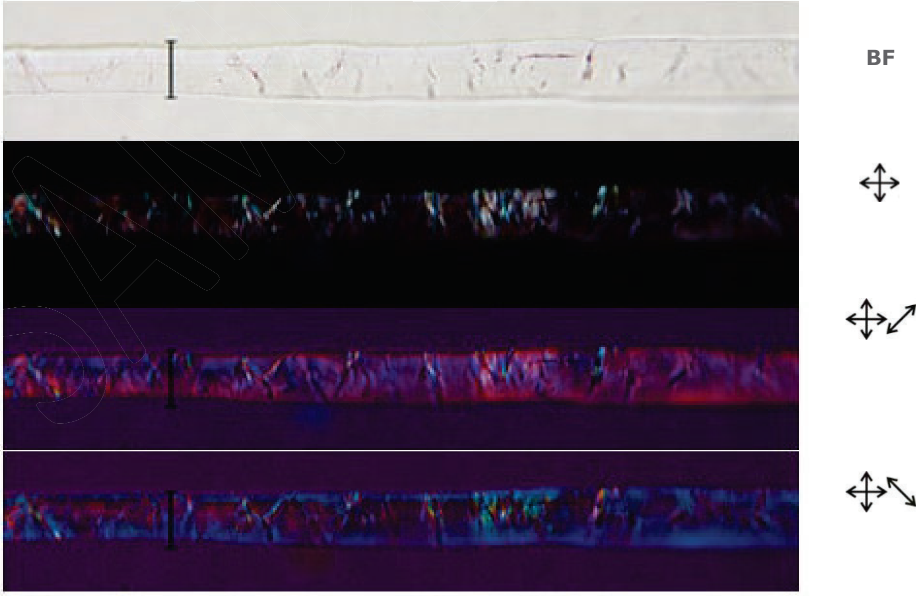
Ph. 3-50 Mercerized cotton viewed in bright field and with polarized light.
Magnification 200x (I = 50 μm)

Kapok is a hollow fibre with a thin cell wall. Between crossed polars, kapok produces first-order milky white and grey colours, as shown in **[Ph. 3-51]**. There is a clear difference in the optical path between the centre and sides of the fibre. When adding or subtracting an order using a lambda plate, blue and red polarization colours appear; these resemble the interference colours of low birefringent fibres with a positive elongation. However, brighter blue and red occurs where more cell wall is present.

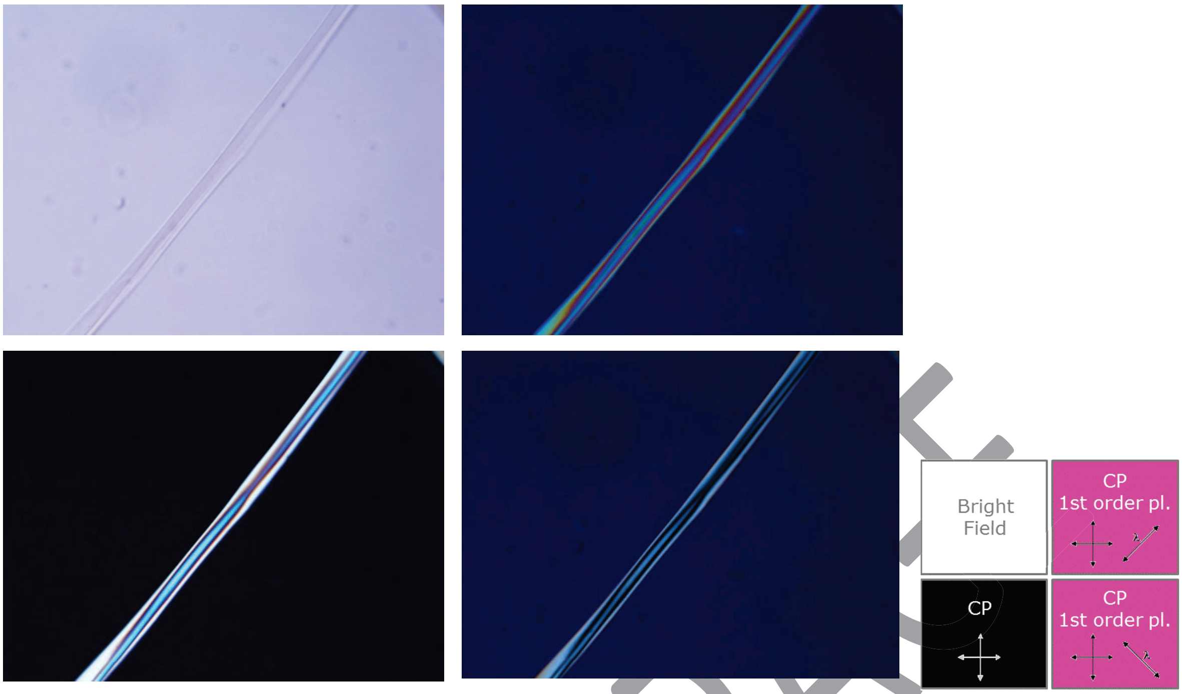


Ph. 3-53 Modified Herzog test on hemp: Z twist.
Magnification 640x (I = 16 µm)

Ramie, jute and kenaf are other bast fibres. Between crossed polars, these fibres present diffuse zones of first-order grey colours. An example of their modified Herzog tests is presented respectively in **[Ph. 3-54]**, **[Ph. 3-55]** and **[Ph. 3-56]**.



Ph. 3-54 Modified Herzog test on ramie: S twist.
Magnification 640x (I = 16 µm)



Ph. 3-61 Silk viewed in bright field and with polarized light.

3.5.4 Observation of dichroism

Of course, the procedure for observing dichroism in man-made fibres also applies to natural fibres. The main difference is that most natural fibres are not uniformly dyed, which complicates the observation of a weak dichroic effect. In **[Tab. 3-4]** some data on dichroic effects present in the most abundant natural fibre classes is shown.

Tab. 3-4 Dichroic effects occurring in some natural fibre classes.

Fibre class	Strong	Weak	None	Number of samples
Dyed cotton fibres (CO)	45%	33%	22%	199
Dyed wool fibres (WO)	0%	0%	100%	650
Dyed silk fibres (SE)	6%	37%	57%	70

In general, a weaker dichroic effect is noted for natural fibres than for man-made fibres having a similar chemical composition. For instance, in viscose, strong dichroic effects are very common (93%), while in cotton, strong dichroism is noted in only 45% of the samples. Similarly, for nylon fibres, a strong or weak effect is observed in 80% of the samples. Animal fibres are protein structures with a polypeptide backbone consisting of amide bonds, such as the keratine in wool or the fibroin in silk. In wool, dichroic effects have not yet been observed. Only 43% of the silk samples showed some dichroic effect,

Tab. 3-6 Generic fibre classes with overlapping birefringence ranges.

Polarization colours resemble	Birefringence range (x 10^3)	Possible confusion with	Birefringence range (x 10^3)
Acrylics (PAN)	-1 → -3	Modacrylics	-2 → 2
		Triacetate	-1 → 0
Polyamide (PA)	49 → 63	Polyethylene	30 → 52
		Polyester, PTT	60 → 80
Polyester (PES)	147 → 175	Unknown	
Viscose (Visc)	20 → 28	Cupro	21 → 37
		Polypropylene (PP)	28 → 34
		Polylactic acid (PLA)	28 → 30

A qualitative description of the polarization colours could consist of the following list:

- no polarization colours (isotropic);
- first-order grey colours, negative elongation;
- first-order grey colours, positive elongation;
- first-order colours;
- bright 2nd to 3rd order colours;
- pale higher-order colours;
- undetermined.

The last description is used whenever no polarization colours can be observed due to the small fibre diameter or when the colours are masked due to deep dyeing.

For fibre comparison, it may be a good idea to note the colour of the central polarization band together with the estimated thickness at the central point. From these data, an estimate of the birefringence can be obtained.

3.6.6 Robustness

In theory, interference colours should be observed under a 45° orientation of the fibre. A small deviation of ± 10° has no noticeable influence on the observed polarization colours, as illustrated for the nylon sample in **[Ph. 3-65]**. If the orientation is under 60° or more, the interference colours become markedly less bright. If qualitative polarization microscopy is performed, the fibre should be brought only approximately in the diagonal position. However, if one is performing quantitative polarization microscopy, an exact positioning under 45° is necessary for a correct determination of the birefringence.