TECHNICAL REPORT

ISO/TR 11827

First edition
2012-06-01

Textiles — Composition testing — Identification of fibres

Textiles — Essai de composition — Identification des fibres

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Reference number
ISO/TR 11827:2012(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TR 11827 was prepared by Technical Committee ISO/TC 38, *Textiles.*

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Introduction

The correct identification of fibres in textiles and the accurate determination of the composition of each fibre present is a legal requirement in many countries throughout the world for imported textile goods and at the point of sale to the public. Fibre identification can be carried out by a number of different techniques, e.g. microscopy, solubility, spectroscopy, melting point, pyrolysis, density, refractive index, etc.

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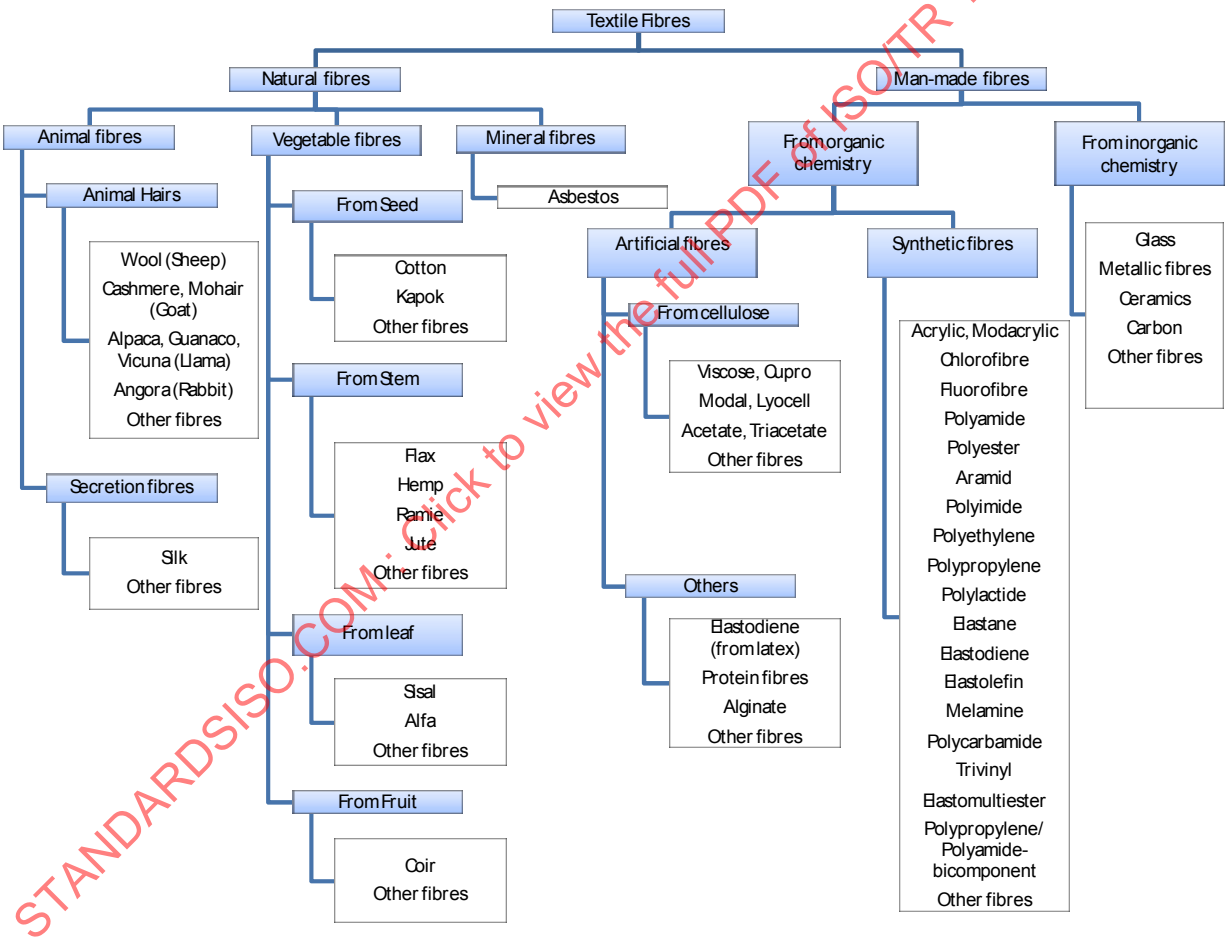
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Textiles — Composition testing — Identification of fibres

IMPORTANT — The electronic file of this document contains colours which are considered to be useful for the correct understanding of the document. Users should therefore consider printing this document using a colour printer.

1 Scope

This Technical Report describes procedures for the identification of natural and man-made fibres, and may be used, when necessary, to coordinate with methods for the quantitative analysis of fibre blends.



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Figure 1 — Classification of the textile fibres in relation to their origin

2 Safety note

This Technical Report calls for the use of substances/procedures that may be injurious to the health/environment if appropriate conditions are not observed. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety/environment at any stage.

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3 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1833-4, *Textiles — Quantitative chemical analysis — Part 4: Mixtures of certain protein and certain other fibres (method using hypochlorite)*

ISO 2076, *Textiles — Man-made fibres — Generic names*

ISO 6938, *Textiles — Natural fibres — Generic names and definitions*

4 Terms and definitions

For the purposes of this document, the following terms and definitions given in ISO 2076 and ISO 6938 and the following apply.

4.1
natural fibre
fibre which occurs in nature: it can be categorized according to its origin into animal, vegetable and mineral fibre

4.2
man-made fibre
manufactured fibre
fibre obtained by a manufacturing process

4.2.1
artificial fibre
manufactured fibre made by transformation of natural polymers (macromolecular material existing in nature)

4.2.2
synthetic fibre
manufactured fibre made from synthetic polymers (macromolecular material which has been chemically synthesised)

4.2.3
bicomponent fibre
fibre composed of two fibres forming polymer components, which are chemically or physically different or both

5 Principle

Objective: identify the fibres

Means: based on fibre properties (single or combination)

Properties for example:

- Morphology
- Solubility
- Light absorption or transmission by IR
- Burning behaviour
- Thermal behaviour

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- Colouration
- Optical behaviour
- Elemental composition

6 Apparatus and preparation of solutions

6.1 Apparatus

6.1.1 **Light Microscope,** using transmitted light

6.1.2 Scanning Electron Microscope

6.1.3 Bunsen Burner or other flame source

6.1.4 Infrared Spectrometer

6.1.4.1 Attenuated Total Reflection (ATR) spectroscopy device

6.1.4.2 Fourier Transform Infrared (FT-IR) spectrometer

6.1.5 Melting Point device (heated block)

6.1.6 Differential Scanning Calorimeter (DSC)

6.1.7 Thermal Gravimetric Analysis (TGA) device (thermobalance)

6.1.8 Gravimetric device (density gradient column)

6.1.9 Energy Dispersive X-ray (EDX) device

6.2 Preparation of solutions

Use only reagents of recognized analytical grade.

6.2.1 Sodium hydroxide and calcium oxide

Prepare a mixture of sodium hydroxide and calcium oxide (mass ratio of 1:1,4)

6.2.2 Iodine/potassium iodine solution

Dissolve 20 g of potassium iodide in 20 ml to 50 ml of distilled water. In this solution dissolve 2,5 g of iodine and dilute to 100 ml

6.2.3 Zinc chloride/iodine solution

Dissolve 66 g of zinc chloride, anhydrous, and 6 g of potassium iodide in 34 ml of water.

Add a small amount of iodine crystal so that the solution is saturated.

6.2.4 Chlorine bleaching solution

Prepare the solution according to ISO 1833-4.

6.2.5 Zinc chloride/formic acid solution

Dissolve 100 g of zinc chloride, anhydrous in 100 ml of water.

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Set the density of this solution to 1,566 g/ml.

Add 6 ml of concentrated formic acid to 100 ml of this solution.

6.2.6 Sodium carbonate 0,25 % solution

Add 0,25 g of sodium carbonate to 100 ml of distilled water and dissolve.

6.2.7 Sodium hydroxide 5 % solution

Dissolve 5 g of sodium hydroxide in distilled water and dilute to 100 ml.

6.2.8 Sulphuric acid 75 % solution

Add carefully, while cooling, 700 ml of concentrated sulphuric acid (ρ 1,84 g/ml) to 350 ml of distilled water.

After the solution has cooled to room temperature, dilute to 1 l with water.

6.2.9 Chloroform/trichloroacetic acid solution

Dissolve 50 g of trichloroacetic acid in 50 g of chloroform.

6.2.10 Ethanol / potassium hydroxide solution

Dissolve 15 g of potassium hydroxide in 100 ml of ethanol.

7 Techniques

7.1 Microscopy

7.1.1 Light Microscopy

Examine the longitudinal view and/or the cross section of a fibre sample under a light microscope (6.1.1) using transmitted light and magnification.

Compare with photomicrographs in Annex B.

7.1.2 Scanning Electron Microscopy

Examine the longitudinal view and/or the cross section of the surface of a fibre sample under a scanning electron microscope (6.1.2) using magnification.

Compare with photomicrographs in Annex C.

7.1.3 Refractive Index

7.1.3.1 General

Refractive index governs the visibility of all colourless and transparent objects.

When a fibre is examined in air (n=1,0), the relatively large difference in refractive index between the fibre and air causes about 5 % of the incident light to be reflected and the transmitted light to be markedly refracted. These effects produce heavy shadows that obscure fine details of the fibre structure and can introduce misleading identification. To reduce the degree of contrast in the shadow regions the fibres are mounted in a medium of suitable refractive index for microscopic evaluation.

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7.1.3.2 Mounting media

If fibres are mounted in a medium of similar refractive index, surface characteristics are practically invisible but internal structure and the presence of voids, or inclusions such as pigmentation, are clearly revealed. When it is desired to examine the surface details of a fibre a mounting medium of significantly different refractive index should be selected, preferably one with a much higher refractive index than that of the fibre, e.g. 1-bromonapthalene or di-iodo-methane.

Mountants should be relatively stable, and should not be volatile or react with the polymer fibre. The most commonly used mountant is liquid paraffin which gives an image of satisfactory contrast for all fibres except for cellulose diacetate and triacetate, for which n-decane is recommended.

It is recommended that all fibres be examined as soon after mounting as possible. Some fibres if left for a period may be penetrated by the mountant, or they may swell which makes fibre diameter measurements incorrect, or the mountant may evaporate.

7.1.3.3 Factors governing refractive indices

Factors governing the refractive index of fibres are the chemical nature of the molecules, the physical arrangement of these molecules, the wavelength of incident light, moisture content, and other substances that may be present in the fibre. In order to make accurate determinations it is necessary to use plane-polarised light under conditions of controlled temperature and relative humidity.

Birefringent substances exhibit different indices of refraction for a given wavelength depending on the direction of light passing through them, as well as upon its direction of transmission. For positive birefringent fibres the maximum and minimum refractive index corresponds to the long axis of the fibres and at right angles to the axis respectively. For negative birefringent fibres the reverse occurs.

7.1.3.4 Behaviour under polarised light

Determination of the behaviour under polarised light of a fibre can be carried out by mounting the fibre in a mountant of known refractive index (Table 2), then viewing under polarised light such that the microscope provides light polarised in the 6-12 o'clock direction.

Align the fibre in the direction of the light and set the microscope to provide axial illumination. Focussing carefully on the outlines of the fibre adjust the focus to just above the fibre. For cylindrical fibres, if the refractive index is higher than that of the mountant the fibre will act like a lens and a bright line of light will move into the middle of the fibre as the focus is raised. If the refractive index is lower that that of the mountant the light will flare out as the focus is raised and the middle of the fibre will become darker.

The test works best on round fibres, for flat ribbons it may be easier to see movement of a bright line at the outlines of the fibre.

Rotating the specimen 45° and setting the microscope to provide cross polars allows birefringence to be seen. Record if the fibre appears very bright (strong birefringence), dim (weak birefringence), or dark (no birefringence).

Repeat the test using different mountants (see Table 2). As the refractive index of the liquid approaches that of the fibre the fibre becomes less distinct until almost invisible. From the table match the liquid to the fibre for identification. This technique is particularly useful for the identification of polyester.

Compare the observations made with the Table 1 to identify possible fibres.

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Table 1 — Refractive Indices of Fibres (cf. [1])

Fibre		Refractive Index		Birefringence	
		Long $n_{//}$	Cross $n_{\perp}$	Δn	
Acetate	Diacetate	1,476	1,473	0,003	Weak
	Triacetate	1,469	1,469	0	Weak
Acrylic		1,511	1,514	-0,003	Weak, negative
Aramid	(Para-)aramid	>2,000	-	-	-
Asbestos	Chrysotile	1,50 - 1,56	-	varies	Strong
	Amosite	1,64 – 1,69	-	varies	-
	Crocidolite	1,68 – 1,71	-	varies	-
Chlorofibre		1,541	1,536	0,005	Weak
Cupro		1,553	1,519	0,034	Strong
Glass		1,52 – 1,55	-	-	None
Modacrylic		1,52 – 1,54	1,52 – 1,53	0,002 – 0,004	Weak
Polyamide	Polyamide 11	1,553	1,507	0,046	Strong
	Polyamide 6	1,575	1,526	0,049	Strong
	Polyamide 6.6	1,578	1,522	0,056	Strong
Polyester		1,706	1,546	0,160	Intense
Polyolefin	Polypropylene	1,530	1,496	0,034	Strong
	Polyethylene	1,574	1,522	0,052	Strong
Viscose		1,54 – 1,55	1,51 – 1,52	0,02 2– 0,039	Strong
Wool		1,557	1,547	0,010	Weak
Cotton		1,577	1,529	0,048	Strong
Silk	Degummed	1,591	1,538	0,053	Strong
Flax		1,58 – 1,60	1,52 – 1,53	0,06	Strong

Table 2 — Refractive Indices of Mountants for Microscopy (cf. [1])

Mountant	Refractive Index
Water	1,33
n-Heptane	1,39
Silicone Fluid (200/100,000cs)	1,406
n-Decane	1,41
Butyl stearate	1,445
Liquid Paraffin	1,47
Olive oil	1,48
Cedarwood oil[a]	1,513-1,519
Anisole	1,515
Ethyl Salicylate	1,525
Methyl Salicylate	1,537
o-Dichlorobenzene	1,549
Bromobenzene	1,56
1-Bromonaphthalene	1,658
Di-iodo-methane (Methylene iodide)	1,74
[a] refractive index of cedarwood oil changes with time	

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7.2 Flame tests

7.2.1 Burning Test

Burning fibres and assessing the characteristics of the flame and fumes given off is a classical method of identifying a class of fibre, such as cellulosic, protein, synthetic, etc.

Present the sample, where possible, to the flame of a Bunsen burner (6.1.3) in the same physical state, e.g. as a twisted thread, to minimise burning differences due to the physical nature of the sample

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Characteristics such as melting or shrinking from the flame should be noted. If the sample burns it should be removed from the flame to see if it continues to burn. The nature of the residue or the odour should also be noted.

Care must be taken in interpreting results where a mixture of fibres is present as one fibre type may mask the presence of another. Also, the presence of finishes or coatings may give misleading results.

Results of the reaction of fibres to flame can be found in Annex A.

7.2.2 Chlorine detection test

Heat a copper wire in a Bunsen burner flame (6.1.3) until any green colouration disappears.

Remove the wire from the flame and touch the fibre with the hot end so that some adheres to it.

Again introduce the wire into the flame. The presence of chlorine in the fibre is indicated by green colour in the flame.

NOTE 1 Chlorine containing fibres - chlorofibre, polyvinylidene and modacrylic fibres.

NOTE 2 Chlorine detection test is called “Beilstein test”.

7.2.3 Nitrogen detection test

Put a few fibres (approximately 100 mg has been found suitable) into a test tube and cover with soda lime or a mixture of sodium hydroxide and calcium oxide (6.2.1) and heat the bottom of the test tube.

NOTE 1 A piece of cotton pad can be inserted in the tube in order to avoid any spitting.

When exposed at the opening of the tube, a wet red litmus paper will change to blue if the fibre contains nitrogen component.

NOTE 2 Nitrogen-containing fibres: silk, wool and animal hairs, polyamide, acrylic, modacrylic, elastane, aramid and melamine fibres.

7.3 Staining Tests

7.3.1 Colouration test with iodine/ potassium iodide solution

Observe the colouration of a fibre sample after immersion of the sample into iodine/ potassium iodide solution (6.2.2) for 30 to 60 seconds and then washing it, and compare the observation with that in Annex A.

7.3.2 Xanthoproteic reaction

Detect protein components in a fibre.

Drop nitric acid onto a fibre sample on a slide glass under a microscope and observe the colour of the fibre. In case yellow colour appears and it changes to orange with neutralization by ammonium, the fibre is composed of proteins.

NOTE Silk, wool and animal hairs, and protein fibre will come under this category.

7.4 Solubility Tests

7.4.1 Polyester confirmation

In the light microscope preparation add some drops of ethanol / potassium hydroxide solution (6.2.10) to the fibres (don’t use immersion oil or other fluid). Warm up slightly, observe in light microscope (6.1.1). Polyester fibres changes morphologically (« hair » grows in the surface of the fibres).

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7.4.2 Cellulose confirmation

Under light microscope (6.1.1), add some drops of copper (II) ethylenediamine reagent to the fibres. Cellulosic fibres are dissolved by this solution.

Compare with data on fibre solubility in Annex D.

7.5 Infrared Spectroscopy

7.5.1 General

The identification of polymers in general and synthetic fibres in particular can be achieved readily by this technique, which provides an instrumental alternative to the classical tests: microscopy, solubility, and staining tests. One great advantage of infrared examination is that the spectrum obtained is determined mainly by the chemical constitution of the fibre and is, in general, less dependent on physical structure, variations in which can affect the results obtained from staining, solubility, and other physical tests used for fibre identification. Where only a few milligrams of sample are available, infrared spectroscopy is probably the most valuable single test. The method is particularly useful with synthetic fibres such as polyolefin, aramids and acrylic fibres, especially the latter, where the constitution and proportion of the acrylonitrile comonomer used are frequently modified.

NOTE However, if two or more synthetic fibres are derived from the same basic monomer, whose properties have been modified by the addition of the same comonomer in different amounts, and if the percentage difference is small, it may not be possible to distinguish the fibres by qualitative infrared examination. Where the comonomer is different, however, then the infrared spectrum obtained will be specific for that particular fibre.

When infrared radiation is passed through a substance, the energies of the IR photons are sufficient to cause rotations and vibrations of molecules and atomic groups. Certain frequencies are absorbed and others are transmitted depending on the nature of the chemical groups.

The absorption of the IR radiation by organic components consists in two main types of vibrations:

- Elongation vibrations (stretching)
- Deformation vibrations (bending)

Infrared spectroscopy, therefore, consists of determining the frequencies at which absorption occurs and preparing a plot of percentage radiation absorbed against frequency. In practice, this is carried out automatically by the infrared spectrometer (6.1.4).

Infrared absorption spectra are measured either with dispersive double-beam (grating) spectrophotometers or with Fourier transform spectrometers, which give a digital interferogram that is subsequently transformed by a computer into the recognizable infrared spectrum.

The majority of commercial spectrophotometers scan the spectrum from 2 to 15 nm, that is to say from 4000 cm^{-1} to 670 cm^{-1} in wavenumber.

Due to the number and complexity of the absorption bands, the infrared spectrum of a given molecule is characteristic of that compound and may be used for identification. In comparative studies of two substances, therefore, identical infrared spectra denote identical substances.

7.5.2 Procedure

The spectra of relatively simple organic molecules are usually determined with the compound itself or in a medium transparent to infrared radiation. Sample preparation of synthetic fibres is more complicated and, of the several methods available, the final choice will depend on the nature of the fibre, and the individual operator. The more suitable methods of sample preparation are described in detail.

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7.5.2.1 Pressed-disc Technique

In the pressed-disc technique, one can obtain spectra of relatively large particles that are suitable for qualitative identification purposes, by choosing as the matrix a halide whose refractive index closely matches that of the sample. In general, potassium bromide (n_D=1,56) is suitable.

Briefly, the method consists of mixing the finely divided fibre with finely powdered potassium bromide, which is stored in an oven.

In preparing the disc, a few milligrams of the fibre are cut up finely with scissors. A portion of the finely chopped or powdered material is uniformly mixed in an agate mortar with 300 mg to 500 mg of finely powdered potassium bromide and pressed into a small disc about 1 mm thick in a suitable vacuum die under a pressure of about 500 kPa to 750 kPa. Vacuum alone is applied to the die for 2 minutes, then vacuum and press load are applied simultaneously for 2 minutes. Clear pellets have only small absorption bands at 2,9 µm and 6,1 µm owing to moisture.

NOTE It should always be borne in mind that potassium bromide is very hygroscopic and that water-absorption bands, which may be present in spectra run by this method, can lead to wrong identity. The potassium bromide method has the important advantage over mulling techniques that extremely small samples may be analysed.

7.5.2.2 Mulling

This type of sample preparation pertains to solids that do not lend themselves to other methods of preparation. The mulling liquid should be non-volatile and as non-absorbing as possible in the 2 nm to 15 nm region. Nujol, which is highly purified mineral oil, is the most readily available and generally useful mulling liquid. Absorption bands, due to the oil, occur at 3,4 nm, 6,9 nm and 7,3 nm. Mulling agents which are free from absorption in the preceding regions are hexachlorobutadiene and perfluorocarbon oil.

The customary method, whereby the substance is ground to a fine powder from which the mull is prepared, is satisfactory for well-defined crystalline materials, but less satisfactory for textile fibres and inapplicable to viscous, plastic, and rubbery substances. The method described below, as well as being applicable to these relatively intractable substances, is very much faster to operate and the mull is prepared in a single operation. In this method the material is rubbed between ground-glass plates, thus enabling a more powerful abrasive action to be obtained.

The grinding plates are prepared from 5 mm glass plate cut to a convenient size. Pairs of these are ground together with 200-mesh carborundum powder until uniformly rough, then rubbed together using a few drops of Nujol as lubricant until no further glass powder is produced. Minute flat areas with sharp cutting edges are formed on the plates.

Textile yarns or fabrics are cut to short lengths, i.e., about 0,5 mm to 2 mm and these are mulled a little at a time, more yarn and Nujol being added at intervals. Excellent mulls of the toughest fibres can be obtained in a few minutes. In preparing a mull, the intention is to produce a paste of petroleum jelly-like consistency. The correct consistency is judged by appearance, by the drag of the grinding plates, and by the disappearance of such tell-tale signs as rats' tails in the mull that indicate that macroscopic particles are still present. Finally, the plates are separated and the mull is transferred to rock salt plates for infrared measurement.

7.5.2.3 Solvent-cast Films

In general, a solvent-cast film gives a better spectrum than that obtained by dispersing the same fibre in potassium bromide or in a mull. The cast-film method is not as generally applicable as the pressed-disc technique since a suitable solvent must first be selected, and for some fibres there is no such solvent. Further requirements are that the solvent must not react with the fibre and it must leave no residue on evaporation.

If films are cast from a solvent onto a smooth glass surface, the films obtained may produce an interference fringe pattern in the spectrum owing to a high degree of parallelism between their front and back surfaces. The fringes may interfere with the identification of the weaker infrared bands, but the difficulty can be obviated by the simple expedient of using a roughened glass surface. One surface of the film will then be irregular and fringes are not produced.

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An approximately 5 % solution is made by dissolving the fibre in the hot solvent. Sufficient solution to cover an area of about 50 mm x 25 mm is poured on to a level glass plate whose surface has been roughened with 400-500 mesh carborundum. The temperature of the solution should be well below that at which bubbles form, otherwise holes are left in the film. Most of the solvent is evaporated off at a temperature low enough to avoid bubble formation and, when the film has solidified, it is heated to a higher temperature, preferably in vacuum, to remove the remaining solvent.

The film can usually be peeled from the glass plate after lifting an edge with a razor blade; wetting with water sometimes helps if the film sticks.

Most solvents are completely removed by the heating, but, where any solvent remains, it may be removed by Soxhlet extraction or refluxing; for example, dimethylformamide (DMF) is tenaciously held by acrylic fibres but is completely removed by boiling the film for 0,5 h to 1,0 h in water. It is essential with this method of sample preparation that the solvent be completely removed, otherwise absorption bands (principally at 5,98 µm), owing to the retention of the DMF, will be present in the spectrum of the fibre.

7.5.2.4 Melt-cast Films

Melt-cast films of thermoplastic fibres can be prepared by pressing fibres between polytetrafluoroethylene (PTFE) sheets between heated platens in a laboratory hydraulic press. As a general guide the films should be thin enough to be nearly transparent (5 µm to 35 µm).

7.5.2.5 Attenuated Total Reflection (ATR)

ATR spectroscopy is used for the analyses of the surface of materials by the mean of ATR spectroscopy device (6.1.4.1). Since it requires no preparation of the sample, it is much quicker than the previous methods.

The infrared radiation is passed through an infrared transmitting crystal with a high refractive index, allowing the radiation to reflect several times within the ATR element.

The sampling surface is pressed into intimate optical contact with the top surface of the crystal.

The commonest material used for the crystal in ATR attachments is Thallium Bromide-Iodide KRS-5. Typically, a prism may be 5 cm long x 2 cm wide x 4 mm thick, with side angles of 45°. Light enters from the angled side of the prism and the radiation is reflected approximately 25 times before emerging from the crystal.

Fibres and fabrics, which are among the most difficult materials to handle by transmission spectroscopy, have proved to be quite amenable to study by multiple internal reflection spectroscopy since they require no special preparation techniques for the purpose. The word 'multiple' should be emphasized since the nature of the fibre itself results in poor contact and many reflections are needed in order to ensure sufficient absorption. See Figure 2.

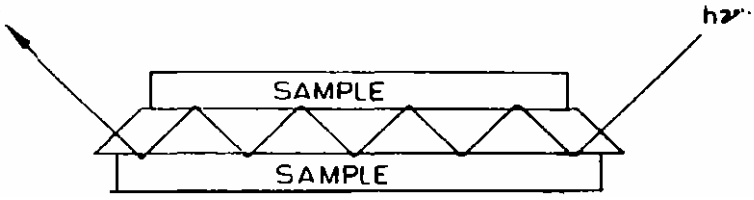


Figure 2 — Multiple internal effect

7.5.2.6 Diffuse Reflectance Spectroscopy

Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) is a newer technique than ATR. Using DRIFT Spectrometer (6.1.4.2), samples can be analysed either directly or as dispersions in non-absorbing matrices, e.g. KBr. A comparison of sampling techniques for the characterisation of cotton textiles showed that the best spectrum was obtained by simply placing a cut circle of fabric in the sample dish.

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When infrared radiation is directed onto the surface of a solid sample two types of reflected radiation can occur. One is specular reflection and the other is diffuse reflection. The specular component is the radiation which reflects directly off the sample surface. Diffuse reflectance is the radiation which penetrates into the sample and then emerges. A diffuse reflectance accessory is designed so that the diffusely reflected energy is optimised and the specular component is minimised. The scattered radiation is collected by a spherical mirror that is focused onto the detector.

7.5.2.7 FT-IR Microscopy

Single fibres can be examined and these are usually flattened with a roller, this being the only destructive part of the technique. Fibres are flattened before analysis to minimise diffusion of radiation, to produce a more uniform thickness (thus minimising deviation from Beer's law and absorption by the fibre) and to increase the sample surface area, thereby enhancing the signal-to-noise ratio while reducing diffraction effects at fibre edges. The minimum sample size is generally of the order of (10 x 10) μm^2.

In the infrared microscope, the sample is mounted on a sample holder (a slide with a 13 mm window or supporting a 13 mm gold reflecting disc). The sample is then brought into focus on the microscope stage using either transmitted or reflected visible illumination. The area of interest on the sample is identified and isolated using adjustable apertures. At this stage, a photograph of the sample may be obtained. An infrared spectrum of the sample is then recorded by switching from visible to infrared radiation using a series of mirrors built into the microscope. The infrared beam penetrates or reflects from the sample and the resultant beam is taken to a highly sensitive detector, which is optimised for the small images generally encountered in FT-IR microscopy.

7.5.3 Interpretation of Spectra

The infrared method depends primarily upon establishing that the spectrum of the unknown matches exactly the spectrum of a known substance examined in the same physical form.

In order to do this, it is necessary to be able to name a compound from the absorption bands it displays in the infrared region.

For example, Table 3 gives several absorption peaks which are characteristic of some main chemical bonds.

Table 3 — Examples of wavenumber of some chemical bonds

Wave number (cm^{-1})	Chemical bond	Chemical family
Around 3 300	O-H	< 3 300 in alcohols > 3 300 in acids
Around 3 250	N-H	Amines, amides
Around 3 000	C-H	2 800 to 3 000 in aliphatic components > 3 000 in aromatic components
Around 2 200	C≡N	Nytril components (Acrylics)
Around 1 700	C=0	Ketones, amides, acids
Around 1 200	C-O-C	Ester components
Around 800	C-Cl	Chloro components

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A laboratory carrying out qualitative analyses customarily sets up its own collection of absorption curves of substances it is likely to encounter. Spectra recorded on the same instrument are to be preferred to literature spectra, because no allowance need be made for differences of resolving power or wavelength calibration. When the spectrum cannot be matched in this way, there is still a possibility that a matching spectrum exists in the literature. A simple method of visual comparison of the unknown spectrum with spectra of known fibres is used in Annex E.

Computerised spectral libraries now exist and most FT-IR software packages incorporate a search routine whereby these commercial libraries or user developed libraries can be accessed.

7.6 Thermal Analysis

7.6.1 Melting Point Determination

If the fibre is made from a thermoplastic polymer it will have a melting point. The melting point can be defined as the temperature (or temperature range) at which crystalline regions melt or the point at which the solid fibre becomes liquid.

Techniques for measuring melting point are usually based on a heated block (6.1.5) for which the temperature can be raised at a variable but controlled rate. Fibres can be placed directly in contact with the block or in a glass capillary tube, the base of which is embedded in a block. If a polarising microscope (or viewer) is used information on crystalline melting can be obtained. Otherwise the temperature at which liquid forms is recorded.

Information on melting points can be found in Annex F.

NOTE Other transition temperatures can also be found in annex F related to non-thermoplastic fibres.

More sophisticated techniques exist (Differential Scanning Calorimetry or Thermal Gravimetric Analysis) but these are usually employed when more detailed information about the fibre is required (see 7.6.2 and 7.6.3).

7.6.2 Differential Scanning Calorimetry (DSC)

7.6.2.1 General

Differential Scanning Calorimetry is an instrumental technique which can be used to study phenomena such as various phase transitions and chemical reactions involving either the absorption or the evolution of heat that may occur when a substance is heated. In the case of fibres, these changes may include the second order or glass transition, desorption of moisture, crystallisation, fusion, chemical reactions and irreversible decomposition processes. For identification purposes, one of the most interesting characteristic is the melting point of a fibre, which can help in distinguishing even fibres classified within the same fibre class, e.g. different types of polyamide (polyamide 6 and polyamide 6.6).

Differential Scanning Calorimetry is a technique in which the difference between the heat flow (power) into a test specimen and that into a reference specimen is measured as a function of temperature and/or time while the test and the reference specimens are subjected to a controlled temperature programme. The result is a curve or thermogram in which temperature or time is plotted on the x-axis and heat flux difference on the y-axis. Peaks in thermogram represent variations from the thermally steady state and correspond to transformations that the sample has undergone. The nature of the measurements makes it possible to distinguish endothermic and exothermic changes; the first ones are usually plotted in downward direction with exotherms shown as upward deflections.

The instrumentation (6.1.6) used for measurement can have two different designs: power-compensation DSC and heat-flux DSC. In the first case, the temperature of both test and reference specimens is kept equal and the difference between the heat flux into the two specimens is measured as a function of temperature or time while varying the temperature of the specimens in accordance with a controlled programme. In the second case, the difference in temperature between the test and reference specimen is proportional to the difference in heat flux which is measured as a function of temperature or time while varying the temperature of the specimens in accordance with a controlled programme.

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It has been shown, in some conditions, that for textile materials, as well as for other materials, the endothermic and exothermic changes observed are both reproducible and uniquely characteristic for a given material, so that DSC curve constitutes a fingerprint and may be used for identification purposes. For this aim, DSC can also be used in the case of fibre mixtures, provided that the melting points of the various constituents the mixture are sufficiently separated.

7.6.2.2 Procedure

7.6.2.2.1 Temperature range

The temperature range selected depends somewhat on the fibre type and fibre history, but, in general, the range from about 25 °C to about 600 °C is most useful. For most synthetic fibres the DSC curve through the fusion point would suffice for identification, whereas, for the natural fibres and cellulosic man-made fibres, identification depends on recording the high-temperature chemical and decomposition reactions.

7.6.2.2.2 Heating rate

Generally, heating rates of 5 °C/min to 10 °C/min (or even 20 °C/min) are satisfactory and permit a useful thermogram to be obtained. Fast heating rates tend to yield sharp major peaks, but if the rate is too fast detail is lost and minor reactions are obscured. Conversely, slow rates of less than 5 °C/min permit small changes to be detected and facilitate reaction separation, but make peak area more difficult to define. Thus, the selection of heating rate is always a compromise. In general, reversible transition temperatures, e.g. melting, are essentially independent of heating rate whereas irreversible reaction peak temperatures are dependent on heating rate. In the latter case, peak temperatures are moved upward by increasing the heating rate. Heating rates need to be carefully specified so that invalid comparisons are avoided.

7.6.2.2.3 Control of atmosphere

Atmosphere is an important variable in the thermal degradation of polymeric materials. To prevent hard-to-control oxidative reactions at elevated temperatures that often produce poorly defined curves, it is recommended that an inert atmosphere, preferably dry nitrogen, be used.

7.6.2.2.4 Sample considerations

In general, the smaller is the sample the better the results. Since a finite time is required for a transformation to occur, a large sample could affect the observed temperature and give rise to a substantial thermal gradient within the sample. In a small sample (1 mg to 10 mg) the thermal gradient is reduced, only semi-micro quantities are required, and volatile degradation products are more readily released. However, care must be exercised since for highly heterogeneous systems a very small sample may not be representative, leading to poor curve reproducibility.

The fibre must be finely divided and excellent results can be obtained when fibre, yarn or fabric samples are cut into 2 mm to 3 mm lengths or into small squares. If low temperature transitions are sought, it is not good practise to grind samples to a powder, because, in the process, sufficient heat can be generated to change the sample history. In general, it is usual practise to run sample as received and if size reduction is required, the gentlest process is used to accomplish the purpose.

7.6.2.2.5 Reference materials

In most instances where sample size is 0,1 mg to 10 mg, no reference material is needed other than the empty pan on the reference side. If a reference material is used, it should be inert and thermostable over the temperature range used. The most commonly used reference material is calcinated alumina, aluminium oxide (Al_2O_3). Two different sample packing techniques can be applied:

- either “sandwich” packing wherein a compressed pellet of ground sample is placed between layers of reference material or
- admixing the sample with reference material as a diluent to yield sample concentration of 5-30 %.

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7.6.2.2.6 Identification

Identification of synthetic fibres can be achieved by

- comparing the curve of the unknown with published curves,
- running an authentic sample under identical conditions for comparison, and
- checking the melting point and other transition data.

7.6.3 Thermal Gravimetric Methods (TGA)

Thermal Gravimetric Analysis (TGA) or thermogravimetry involves the use of the recording thermobalance (6.1.7) to automatically measure and record weight changes which occur when sample of material is heated or cooled to a controlled programme in a controlled environment. The resulting weight change versus temperature curve, or its derivative, gives information concerning the thermal stability and decomposition of the original sample.

7.7 Density measurement methods

The density of fibres is typically determined by the use of a density gradient column (6.1.8) Energy Dispersive X-ray used in conjunction with calibrated glass spheres. Layers of liquids with decreasing density are stacked on top of each other in a graduated cylinder with calibrated glass beads of known density acting as reference material. A fibre of unknown density is placed into the cylinder and then the level at which it comes to rest provides the density of the fibre.

Information on density can be found in Annex G.

7.8 Other Instrumental Methods

7.8.1 Energy Dispersive X-ray (EDX) analysis

Energy dispersive x-ray (EDX) device (6.1.9) is used in conjunction with a scanning electron microscope (6.1.2) to provide a unique capability for analyzing elements present in textile fibres down to lithium in atomic number. The presence of pre-extrusion additives such as titanium dioxide as well as post extrusion additives such as silver can both be assessed. Both semi quantitative values for elemental concentration and mapping capability showing elemental distribution throughout a fibre can be accomplished.

EDX can be used both longitudinally and in cross –section. Depending on the type of scanning electronic microscope used either a carbon or gold sputter coater is often necessary to obtain both high quality images and elemental composition. If a gold coater is used it will mask any gold present in the sample being analyzed and a carbon coater is often a superior choice.

NOTE Coatings and auxiliary chemicals can also be characterized as they are found on textile fibres. Fluorine based finishes as well as flame retardants are examples of elemental coating components that can be revealed using EDX.

EDX is considered to have a limit of detection of about 0,1 % for the portion of the sample that interacts with the analyzing beam. With the use x-ray fluorescence in addition to energy dispersive analysis enables detection of about 0,000 1 % for optimal matrix and element combinations.

EDX is an excellent microanalysis tool with spot size capabilities in the 100 nm range.

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8 Examples of procedures

8.1 Procedure using microscopy, solubility tests and FT-IR tests (examples)

8.1.1 Example n°1

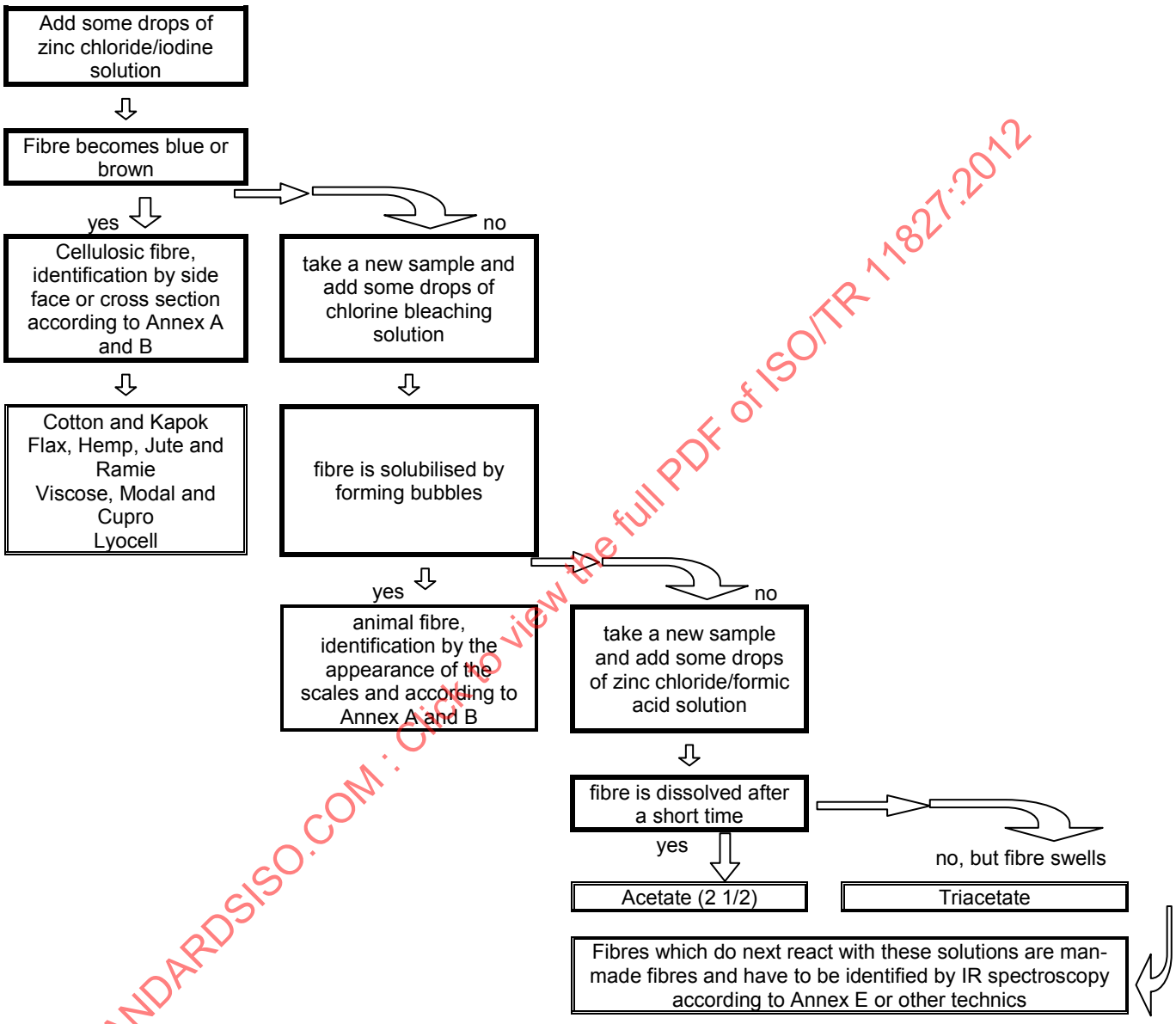


Figure 3 — A procedure using microscopy, solubility tests

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8.1.2 Example n°2

Table 4 — A procedure using microscopy, solubility tests and FT-IR tests

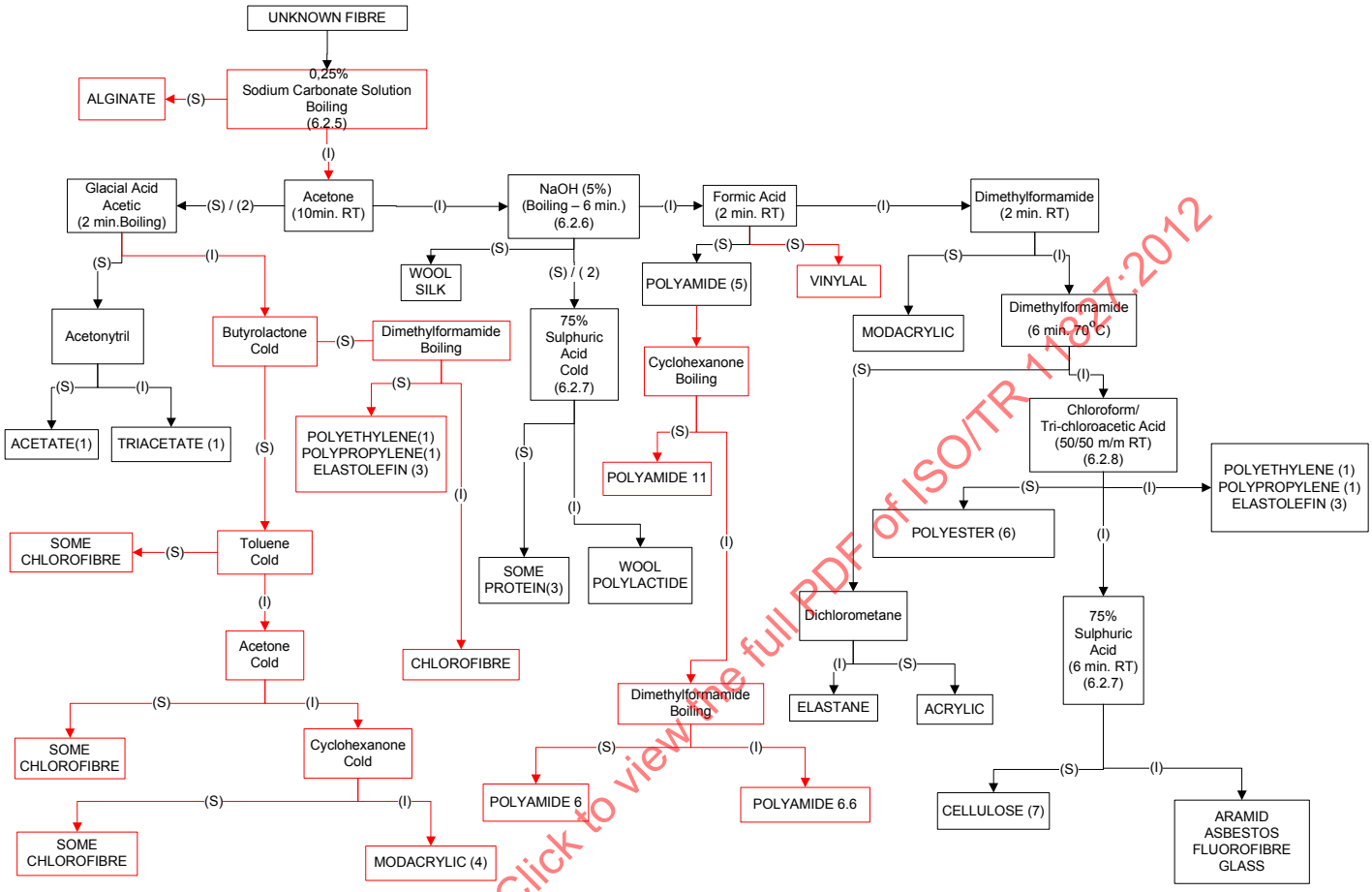
<table>
<tr><td rowspan="3">Sample that contains only one fibre</td><td>Natural fibre</td><td colspan="2">Longitudinal microscopical examination</td></tr>
<tr><td>Artificial or inorganic fibre</td><td colspan="2">Longitudinal microscopical examination</td></tr>
<tr><td>Synthetic fibre</td><td colspan="2">FT-IR spectrophotometer examination</td></tr>
<tr><td rowspan="5">Sample that contains more than one fibre</td><td>Blend of natural fibres with the same chemical composition</td><td colspan="2">Longitudinal microscopical examination (and cross section microscopical examination for blends of liberian fibres; and electronic scanning microscopy examination for wool and speciality fibres)</td></tr>
<tr><td rowspan="3">Blend of chemical synthetic and/or artificial and/or inorganic fibres</td><td>Intimate blend</td><td>Examination through solubility</td></tr>
<tr><td rowspan="2">Yarns/Threads can be manually separated</td><td>FT-IR spectrophotometer examination</td></tr>
<tr><td>Longitudinal microscopical examination (for artificial and/or inorganic fibres)</td></tr>
<tr><td rowspan="3">Blend of (vegetable and/or animal) natural fibres with synthetic and/or artificial and/or inorganic fibres</td><td>Intimate blend</td><td>Examination through solubility (when wool is present, it has to be removed before starting the test)</td></tr>
<tr><td rowspan="2">Yarns/Threads can be manually separated</td><td>Longitudinal microscopical examination (for artificial and/or inorganic fibres)</td></tr>
<tr><td>FT-IR spectrophotometer examination</td></tr>
</table>

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8.2 Procedure using solubility tests (examples)

8.2.1 Example n°1



(S) Soluble ;(I) Insoluble; (RT) Room Temperature

(1) Confirmed by melting point.
(2) Fibre changes morphologically (swells).
(3) should be Confirmed by FT-IR or other technics.
(4) In acetone solution (even boiling) fibre is not dissolved. Fibre changes morphologically.
(5) In the solution of formic acid used to identify the fibre, add twice the volume in cold water. If it is cloudy indicates the presence of nylon fibre.
(6) Microscopic preparation made with ethanol alcohol and potassium hydroxide solution. Warming up slightly, the fibre changes morphologically (“hair grows”). See 7.4.1.
(7) Microscopic preparation made with copper(II) ethylenediamine solution. If is a cellulose fibre, it is dissolved by the solution. See 7.4.2.

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8.2.2 Example n°2

Table 5 — Pre-treatment with sodium hypochlorite for 10 minutes at room temperature in cold bath in case of wool or silk fibres

Steps	Residuals after treatment	Solvent and conditions	Dilution medium	In case of precipitate or flocking presence of ….
1	N[a]	Acetic acid 96 % 10 minutes at room temperature	Water	Acetate (*) or Triacetate (**)
1a (*)	N	Acetone 80 % 5 minutes at room temperature	Water	Acetate
1b (**)	(R)*[b] of 1a	Chloroform 10 minutes at room temperature	Water	Triacetate
2	(R)*	Acetic acid 96 % 2 minutes in boiling waterbath	Water	Polyamide
3	(R)*	Cyclohexane 5 minutes in boiling waterbath	Let it cool	Chlorofibre
4	(R)*	n,n Dimethylformamide 5 minutes in boiling waterbath	Alcoholic Potassium hydroxide and let it warm up	Acrylic , modacrylic fibre or polyamide-imide
5	(R)*	m-Xylene or tetrachloroethylene 2 minutes from boiling solution	Let it cool	Polypropylene or polyethylene
6	(R)*	Phenol 1 minute from boiling solution	Let it cool	Polyester
7	(R)*	Microscopical examination of the residuals and FT-IR spectrophotometer examination (if necessary)		

[a] N = new test specimen

[b] (R)* = residual test specimen of the previous step

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8.3 Procedure using combustion tests and melting point determination (example)

Table 6 — Flame and Combustion tests

Behaviour of the specimen			Fibre type
Approach to Flame	**On Hot Plate**	**In Flame**	
Does not shrink from flame or melt	Chars below 337 °C. Does not melt	Burns with irregular spurting flame, leaving a black, inflated, easily-powered residue, and emits a smell like that of burnt hair[ab]	Protein
		Burns readily, emitting a smell like that of burnt paper, leaving a small amount of ash (or sometimes emitting a distinct fishy odour, leaving a dark, skeletal residue)	Cellulose (or resin treated cellulose)
		Burns slowly, extinguishing when flame removed. Burning may accompanied by evolution of fumes having a pungent odour. Carbonized skeletal residue	Viscose or cellulose with flame Retardant finish
		Burns readily, extinguishing at once on removal from flame, leaving an incandescent residue	Calcium alginate
	Does not char or melt[c]	Melts to a clear hard bead	Glass
		Glows, but retains its original form	Asbestos[d]
Shrinks or melts to a bead	Melts below 337 °C	Burns and drips from the flame	Thermoplastic at low temperature
	Does not melt up to 337 °C	Burns and drips from the flame or chars	Aramid
		Does not burn	Fluorofibre

a Tin-weighed silk shows a skeletal residue that glows in a flame.

b Wool fibre extinguishes when removed from flame

c In the case of glass, smoke may be emitted by dressings and there may be discoloration.

d It should be remembered that asbestos is commonly blended with cotton or some other fibre.

8.4 Procedure using microscopy, FT-IR analysis and thermal analysis, case of bicomponent fibres (examples)

8.4.1 Case of polyethylene / polypropylene bicomponent fibre

8.4.1.1 Analysis by microscopy

The longitudinal view using the light microscopy (Figure 5) shows slightly the configuration Core/Sheath of the bicomponent fibre, although the cross view using the light microscopy (Figure 4) shows the configuration Core/Sheath of this bicomponent fibre.

The cross view using the Scanning Electronic Microscopy (Figure 6) shows the configuration Core/Sheath of the bicomponent fibre, but the longitudinal view using the Scanning Electronic Microscopy (Figure 7) cannot show this configuration.

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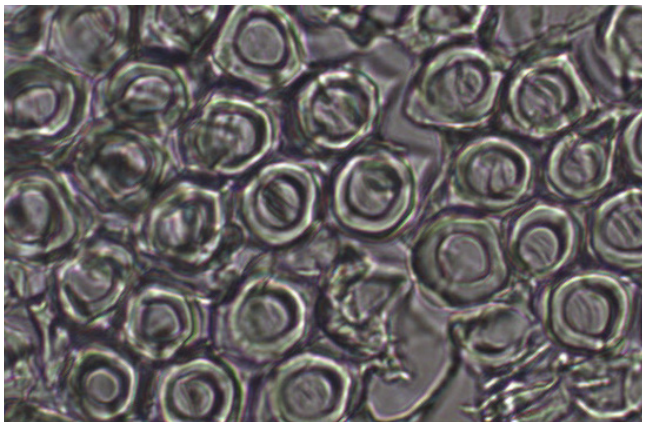


Figure 4 — Light Microscopy - cross view of polyethylene / polypropylene bicomponent fibre

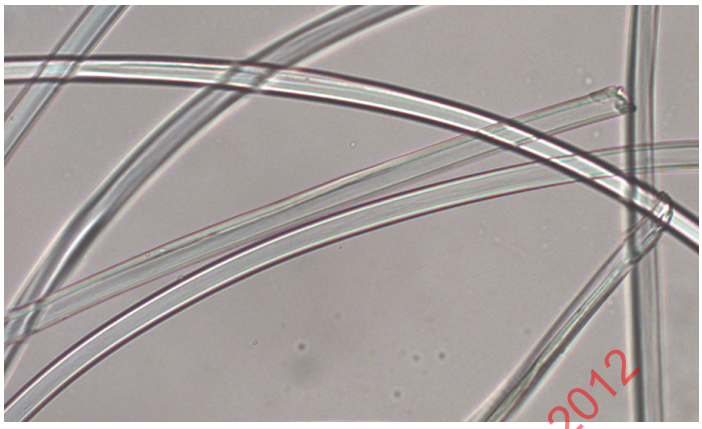


Figure 5 — Light Microscopy longitudinal view of polyethylene / polypropylene bicomponent fibre

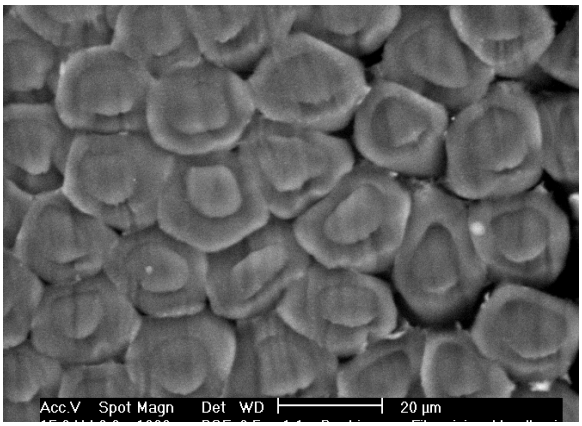


Figure 6 — SEM : cross view of polyethylene / polypropylene bicomponent fibre

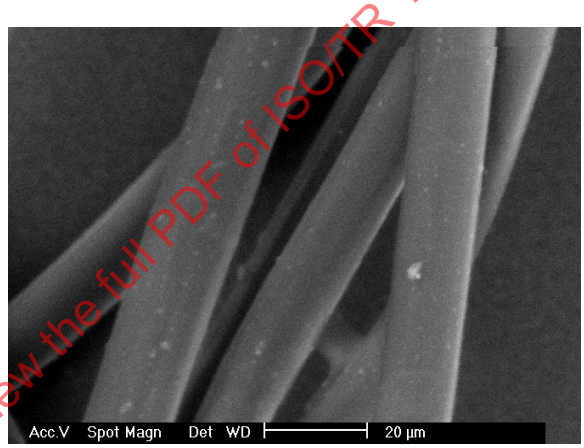


Figure 7 — SEM : longitudinal view of polyethylene / polypropylene bicomponent fibre

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8.4.1.2 FT-IR analysis

The application of the FT-IR analysis along the fibre leads to identify only the presence of polyethylene (Figure 8), although its application across the fibre leads to identify the presence of both polyethylene and polypropylene (Figure 9).

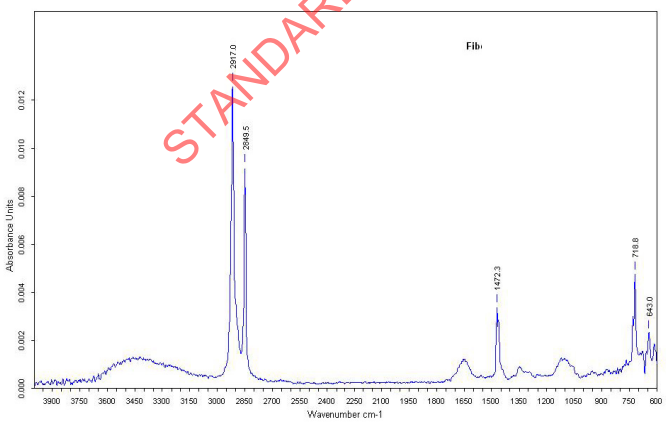


Figure 8 — FT-IR Spectra of polyethylene / polypropylene bicomponent fibre (along the fibre)

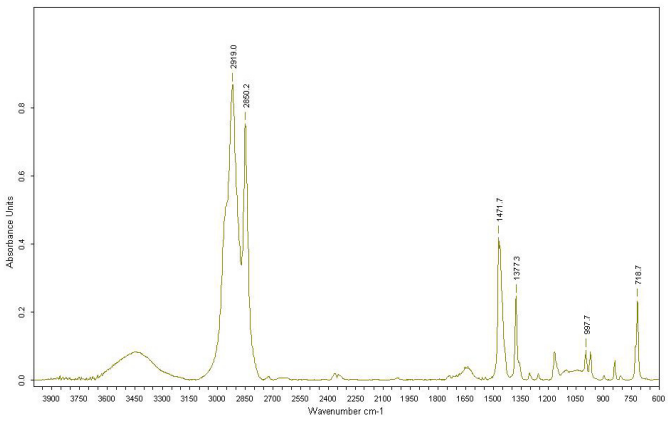


Figure 9 — FT-IR Spectra of polyethylene / polypropylene bicomponent fibre (across the fibre)

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8.4.1.3 Thermal analysis (DSC)

The application of the DSC analysis (Figure 10) shows two peaks related to 2 different materials (during the heating phases).

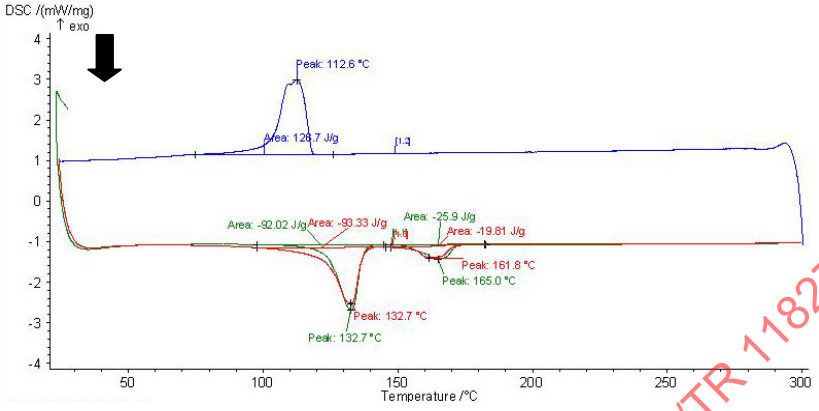


Figure 10 — DSC Spectra of polyethylene / polypropylene bicomponent fibre (across the fibre)

8.4.2 Case of polyester / polyester bicomponent fibre (example)

8.4.2.1 Analysis by microscopy

The longitudinal view using the light microscopy (Figure 11) shows slightly the configuration Core/Sheath of the bicomponent fibre, although the cross view using the light microscopy (Figure 12) shows the configuration Core/Sheath of this bicomponent fibre.

The cross view using the Scanning Electronic Microscopy (Figure 13) shows slightly the configuration Core/Sheath of the bicomponent fibre, but the longitudinal view using the Scanning Electronic Microscopy (Figure 14) cannot show this configuration.

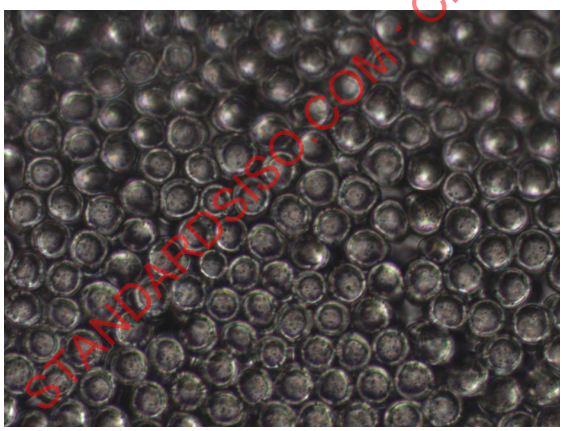


Figure 11 — Light Microscopy - cross view of polyester / polyester bicomponent fibre

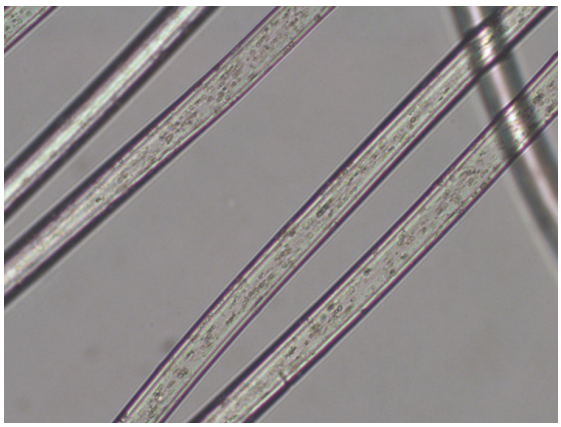


Figure 12 — Light Microscopy longitudinal view of polyester / polyester bicomponent fibre

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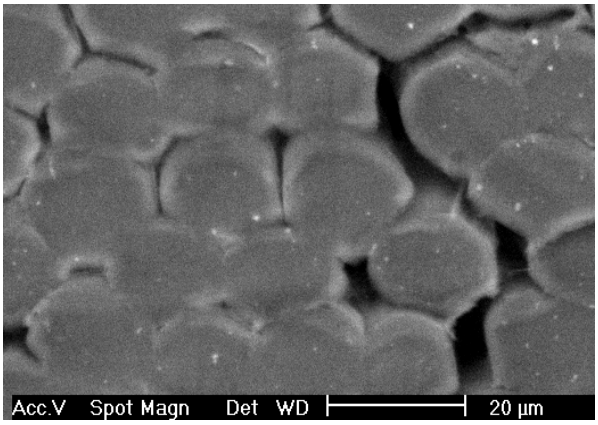


Figure 13 — SEM : cross view of polyester / polyester bicomponent fibre

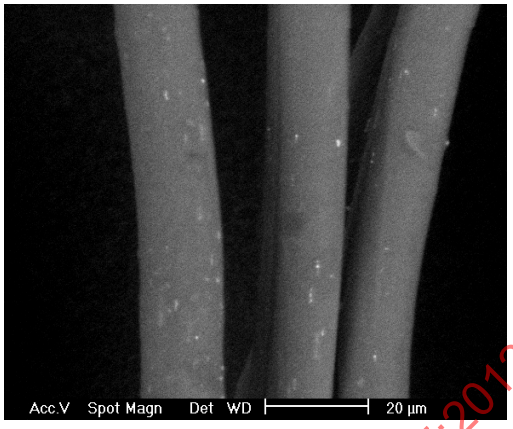


Figure 14 — SEM : longitudinal view of polyester / polyester bicomponent fibre

8.4.2.2 FT-IR analysis

The application of the FT-IR analysis of the fibre leads to identify the presence of polyester (Figure 15).

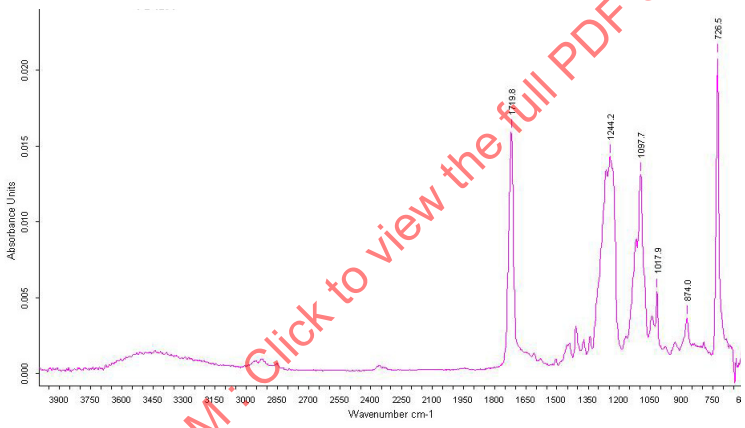


Figure 15 — FT-IR Spectra of polyester / polyester bicomponent fibre

8.4.2.3 Thermal analysis (TGA and DSC)

The application of the TGA method (Figure 16) shows a weight change related to polyester material.

The application of the DSC (Figure 17) shows two peaks related to 2 different materials (during the first heating phases).

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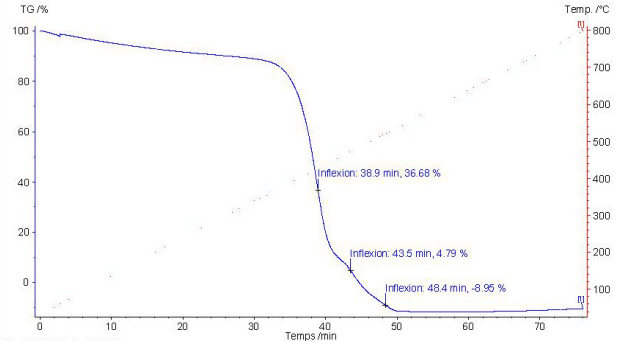


Figure 16 — TGA curve of polyester / polyester bicomponent fibre

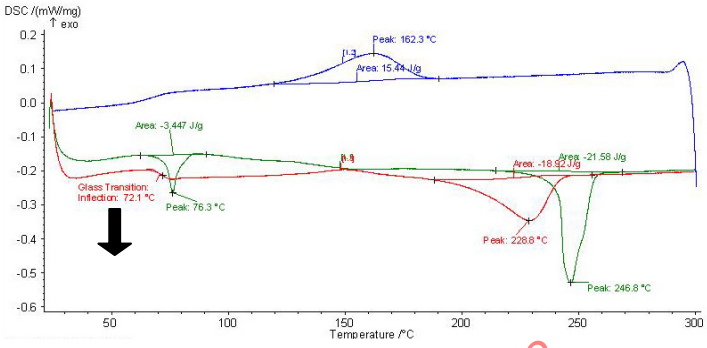


Figure 17 — DSC Spectra of polyester / polyester bicomponent fibre

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Annex A
(informative)

Characteristics relative to fibre identification testing

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Table A.1 — Characteristics relative to fibre identification testing

Name of fibre	Combustion test					Existence of Chlorine	Existence of nitrogen	Microscopic appearance[a]		Colouring by iodine-potassium iodide	Xanthoproteic reaction
	When approaching to flame	In the flame	When separated from flame	Odour	Ash			Side face	Cross section		
Cotton	Burns immediately when touching the flame	Burns	Continuous burning and burns very rapidly, Residual illumination exists	Odour of burning paper	Very small, soft and grey colour	No	No	Flat ribbon state and natural twists over the total length appear (In mercerised cotton these are little)	There are various types such as broad bean type, horse shoe shape, etc, and those having hollow part (mercerised cotton becomes round)	No colouring (mercerised cotton becomes faint blue)	No
Hemp; Flax and Ramie	Same as the above	Same as the above	Same as the above	Same as the above	Same as the above	No	No	Streaks run in fibre axis direction. The top ends of streaks having knots at some places are sharp in flax and obtuse angle in ramie.	The flax is polygon and has hollow part. The ramie is flat elliptic shape and has hollow part.	No colouring	No

[a] The microscopic appearances are described of usual fibres; when the fibre is modified, its appearance is not the same as the description in many cases.

Table A.1 (*continued*)

Name of fibre	Combustion test					Existence of Chlorine	Existence of nitrogen	Microscopic appearance		Colouring by iodine-potassium iodide	Xanthoproteic reaction
	When approaching to flame	In the flame	When separated from flame	Odour	Ash			Side face	Cross section		
Silk	Shrinks and parts from flame	Shrinks and burns	Resembles wool but burns in rather flashing	Odour of burning hairs	Blisters to black, is brittle to be crushed easily	No	Exists	Surface is smooth and there is no change.	Triangle shape	Faint yellow colour	Exists
Wool	Same as the above	Same as the above	Difficul, but continuous burning and prior to burning shrinks	Same as the above	Same as the above	No	Exists	Scale pieces appear	Circular shape in many	Same as the above	Exists
Viscose	Burns immediately when touching the flame	Burns	Continuous burning and burns very rapidly, Residual illumination doesn't exist.	Odour of burning paper	If not dull, ashes don't almost remain.	No	No	Several streaks run in fibre axis direction	The profile is irregular petalous shape.	Black, blue, green colour	No
Cupro and Modal, Lyocell	Same as the above	Same as the above	Same as the above	Same as the above	Same as the above	No	No	Surface is smooth.	Circular shape	Same as the above	No
Acetate	Fuses and parts from the flame	Fuses and burns	While fusing, continuous burning	Acetic acid odour	Black, hard, brittle irregular shape	No	No	One or two streaks run in fibre axis direction	Clover leaf state	Dark brown colour	No
Triacetate	Same as the above	Same as the above	Same as the above	Same as the above	Same as the above	No	No	Same as the above	Same as the above	Same as the above	No
Vinylal	While shrinking fuses	Fuses and burn	Same as the above	Sweet fragrant odour when polyvinyl alcohol burns	Lump state of hard, dark brown irregular shape	No	No	White line running in fibre axis direction apears at the middle part	Existence of cocoon state core layer is confirmed. There is also circular shape.	Thin dark blue colour	No

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Table A.1 (*continued*)

Name of fibre	Combustion test					Existence of Chlorine	Existence of nitrogen	Microscopic appearance		Colouring by iodine-potassium iodide	Xanthoproteic reaction
	When approaching to flame	In the flame	When separated from flame	Odour	Ash			Side face	Cross section		
Polyamide	Fuses	Same as the above	Doesn't continue burning	Odour inherent to amide	Beads from hard dark brown colour to grey colour	No	Exists	The surface is smooth	Circular shape exists in many cases.	Dark brown colour	No
Vinylidene	Shurinks and parts from flame	Fuses and burn with raising smoke. The basic part indicates green colour	Same as the above	Sharp stimulant odour	Blittle, irregular black lump	Exists	No	Same as the above	Same as the above	No colouring	No
Polyvinyl chloride	Same as the above	Fuses and burns rasing black smoke	Same as the above	Resembles vinylidene but is weak	Same as the above	Exists	No	Same as the above	Same as the above	Same as the above	No
Polyester	Fuses	Fuses and burns	Continues burning	Sweet fragrant odour (weak)	Hard, round, black colour	No	No	Same as the above	Same as the above	Same as the above	No
Acrylic	Fuses and catches fire	Same as the above	Burns quickly	Rather resembles the odour when baking meat	Hard, round, black and irregular	(No)[b]	Exists	There are many kinds and not uniform but those of smooth surface are many.	There are circular shapes in many cases but some are heart shape.	Dark brown colour	No
Modacrylic	Shurinks and parts from flame	Fuses and burn with raising black smoke	Doesn't continue burning	Resembles the odour when burning soap	Brittle, iregular, black lump	(Exists)[b]	Exists	One thick line runs in fibre axis direction	Horseshoe shape	Dark brown colour	No
Polypropylene	Same as the above	Fuses and burns gentry with raising smoke	Burns with fusing gently	Resemble the odour when paraffine burns	Hard, grey colour beads	No	No	Surface is smooth	Circular shape	No colouring	No

[b] When ( ) is described, the existence of chlorine is different among types in some cases.

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Table A.1 (*continued*)

Name of fibre	Combustion test					Existence of Chlorine	Existence of nitrogen	Microscopic appearance		Colouring by iodine-potassium iodide	Xanthoproteic reaction
	When approaching to flame	In the flame	When separated from flame	Odour	Ash			Side face	Cross section		
Elastane	Fuses	Fuses and burns	Doesn't continue burning	Particulaly different odour	Rubber state lump having tackiness	No	Exists	Same as the above	There are many kinds and not uniform.	Dark brown colour	No
Aramid (para-) [d]	Red glow but doesn't burn with flame.	Red glow	Red glow disapear	Sweat stimulant odour	Black ashes remain as they are fibrous state.	No	Exists [c]	Surface is smooth, there is no change. In some cases knots are found.	Circular shape	No	No
Aramid (para-) [e]	Red glow and burns with raising flame	Burns	Burns for a while but goes out in a short time.	Sweat sour odour	Black, hard and brittle	No	Exists [c]	Surface is smooth	Circular shape	No	No
Aramid (meta-)	Shrinks and parts from flame	Shrinks and burns	Doesn't continue burning.	Sweat fragrant odour	Black, hard and brittle	No	Exists [c]	Surface is smooth, and a line streak runs in fibre direction.	Near heart shape	No	No
Polylactide	Fuses	Fuses and burns	Fuses gently and burns	Aromatic and a little sweat fragrant odour	Hard, black lump state	No	No	Surface is smooth	Circular shape exists in many cases.	No colouring	No

[c] In the nitrogen detection of aramid fibre, it is necessary to pay attention to recognize the slight change indicating the existence with litmus paper.

[d] this aramid type is made of one diamine and a chloride. This aramid type is broken up into fine segments by sodium hypochlorite.

[e] this aramid type is made of two different diamines and a chloride.

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Annex B
(informative)

Photomicrographs of Fibres (Light Microscopy)

B.1 Monocomponent fibres

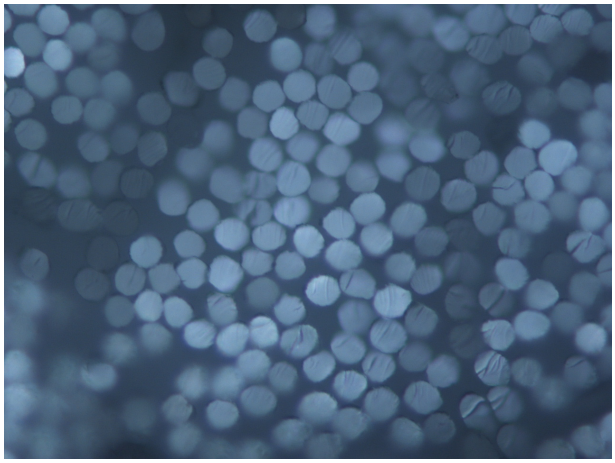


Figure B.1 — Acrylic : cross view

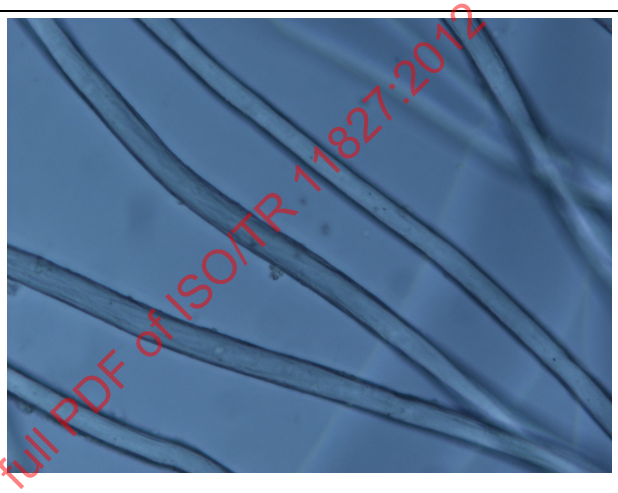


Figure B.2 — Acrylic : longitudinal view



Figure B.3 — Cat hair : longitudinal view

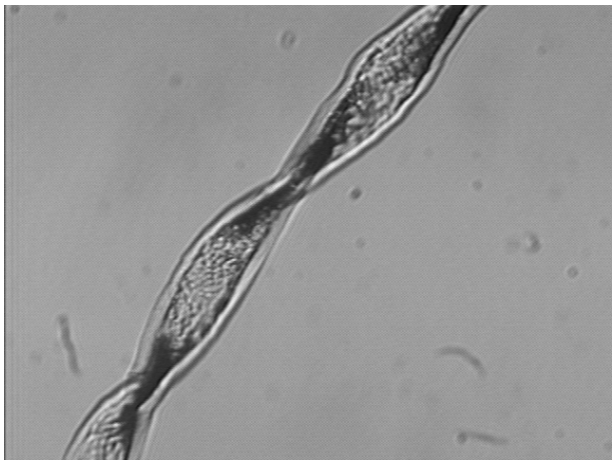


Figure B.4 — Cotton : longitudinal view

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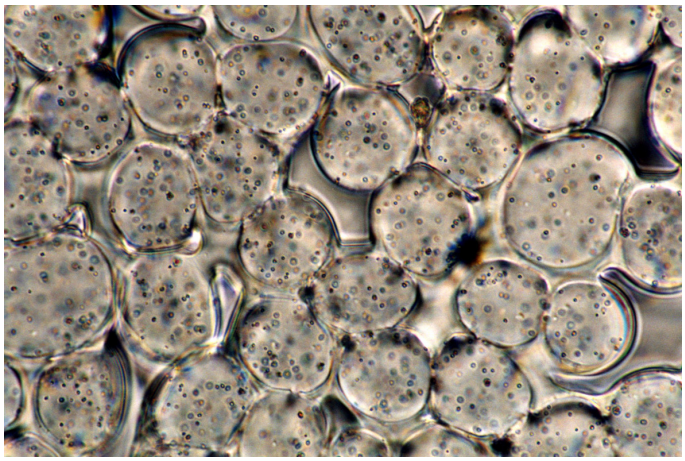


Figure B.5 — Cupro : cross view

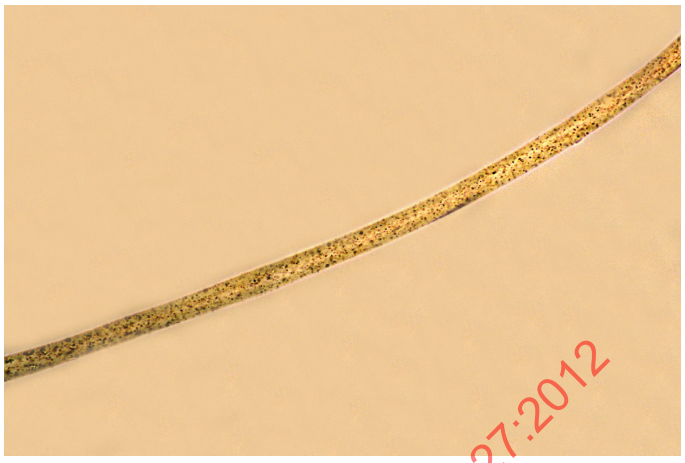


Figure B.6 — Cupro : longitudinal view



Figure B.7 — Elastolefin : cross view



Figure B.8 — Elastolefin : longitudinal view

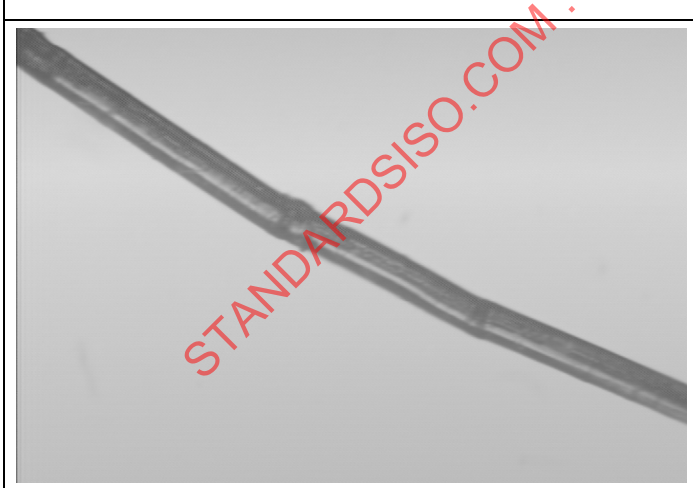


Figure B.9 — Linen (flax): longitudinal view

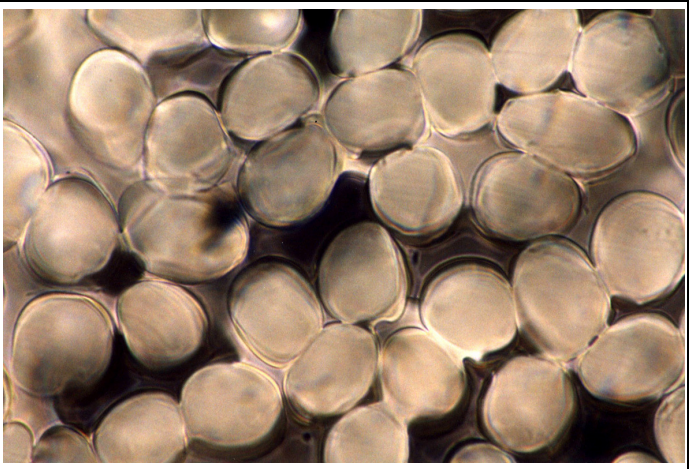


Figure B.10 — Lyocell : cross view

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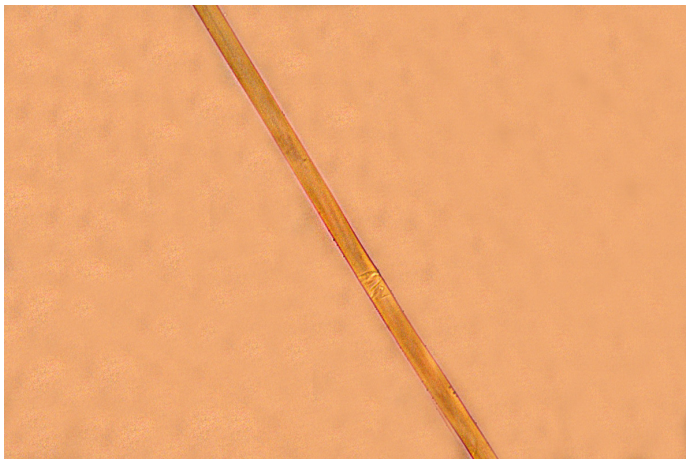


Figure B.11 — Lyocell : longitudinal view

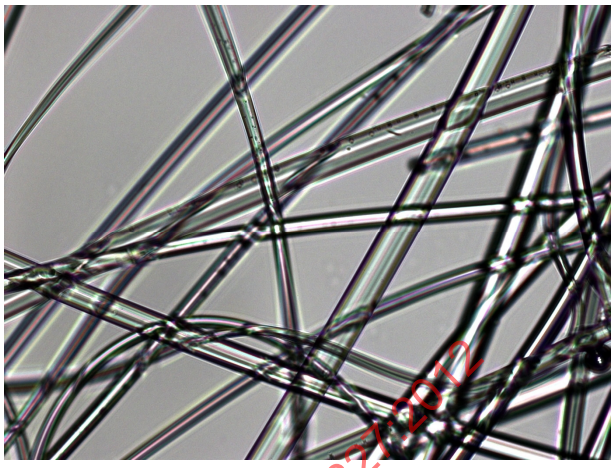


Figure B.12 — melamine : longitudinal view

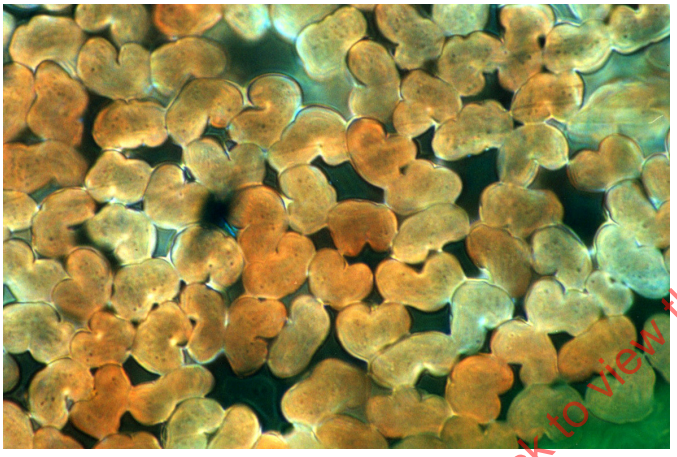


Figure B.13 — Modal : cross view

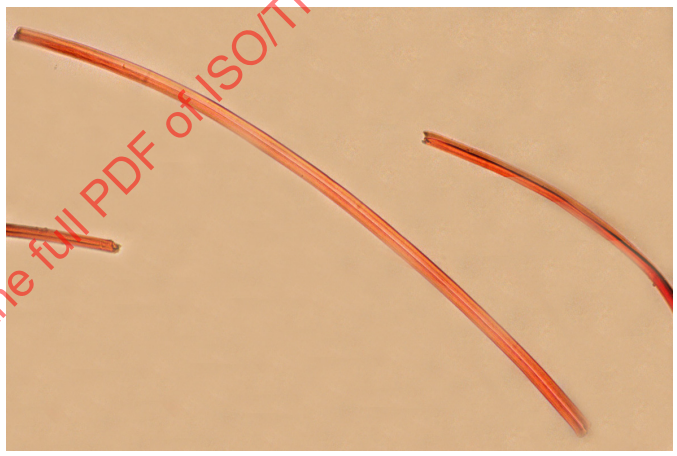


Figure B.14 — Modal : longitudinal view

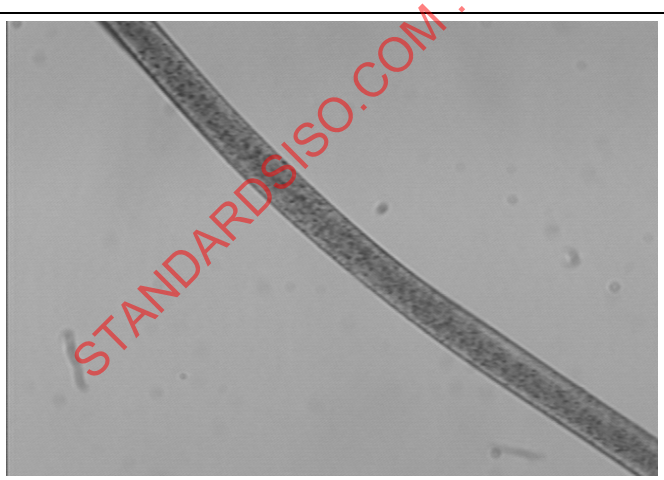


Figure B.15 — Polyamide : longitudinal view



Figure B.16 — Polyester : longitudinal view

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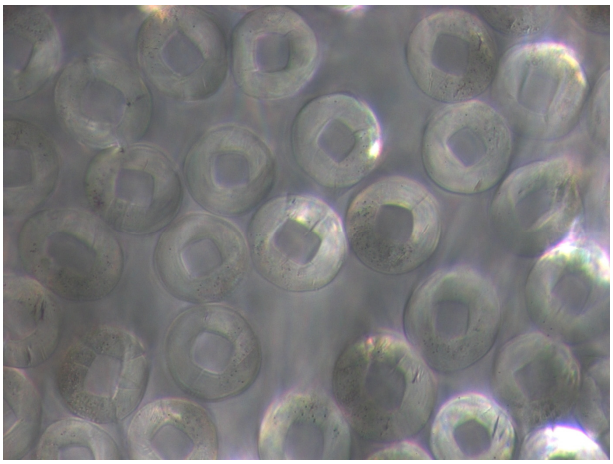


Figure B.17 — polylactide, with hole : cross view

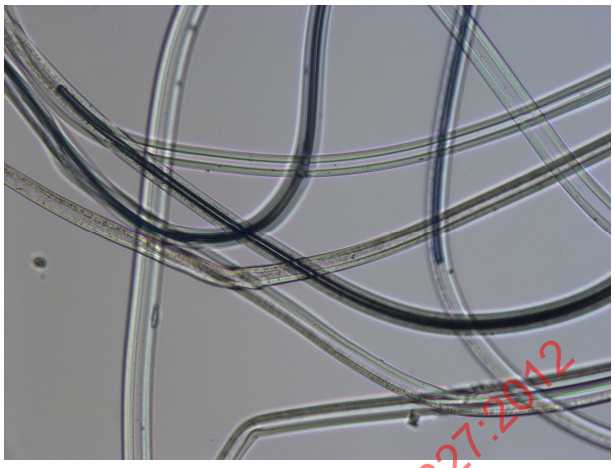


Figure B.18 — polylactide, with hole : longitudinal view

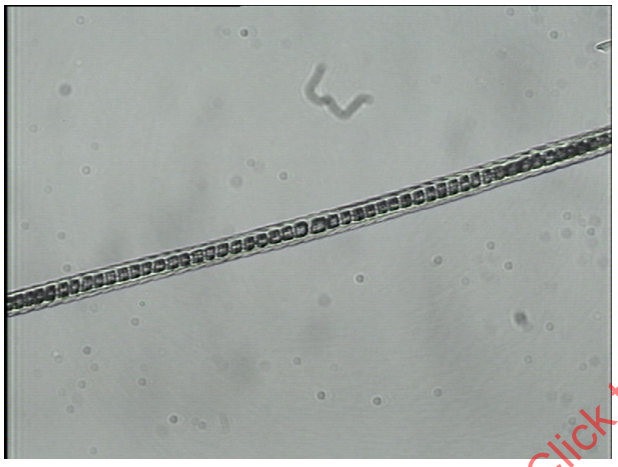


Figure B.19 — Rabbit hair : longitudinal view

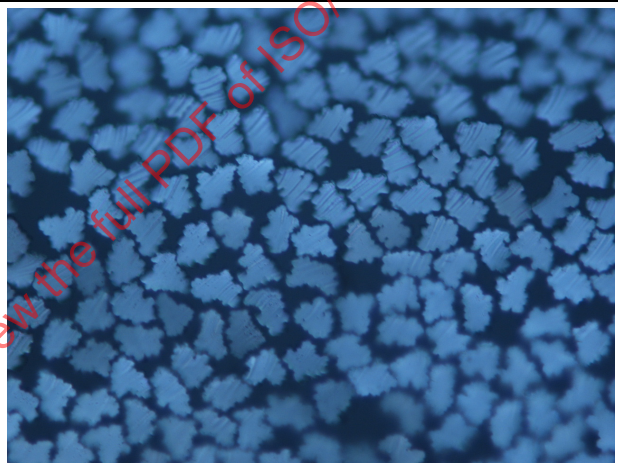


Figure B.20 — Viscose : cross view

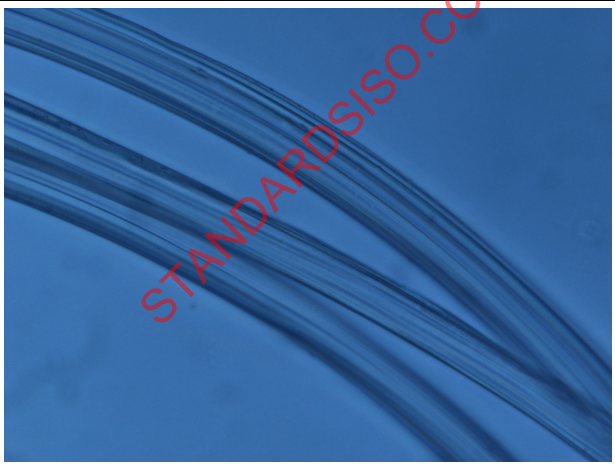


Figure B.21 — Viscose : longitudinal view



Figure B.22 — Wool : longitudinal view

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B.2 Bicomponent fibres

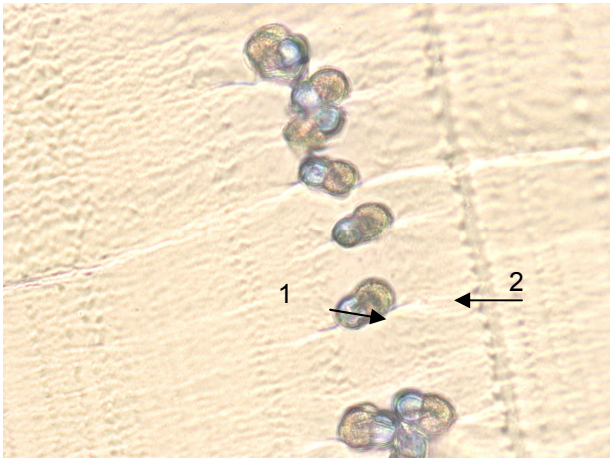


Figure B.23 — elastomultiester : cross view

Key: 1: polyester "a"; 2: polyester "b"

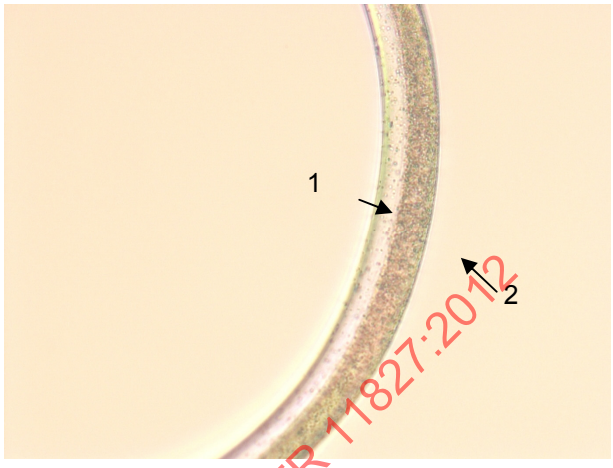


Figure B.24 — elastomultiester : longitudinal view

Key: 1: polyester "a"; 2: polyester "b"

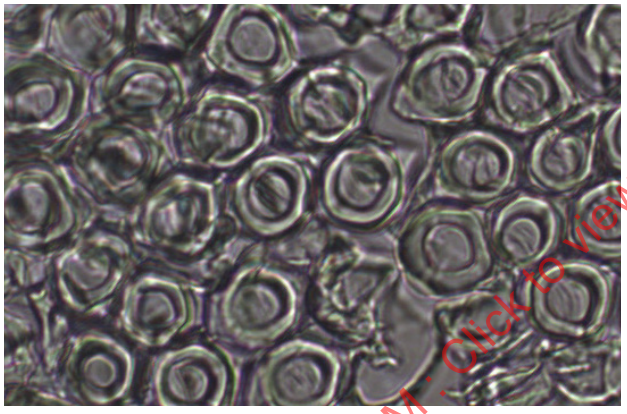


Figure B.25 — Light Microscopy - cross view of polyethylene / polypropylene bicomponent fibre

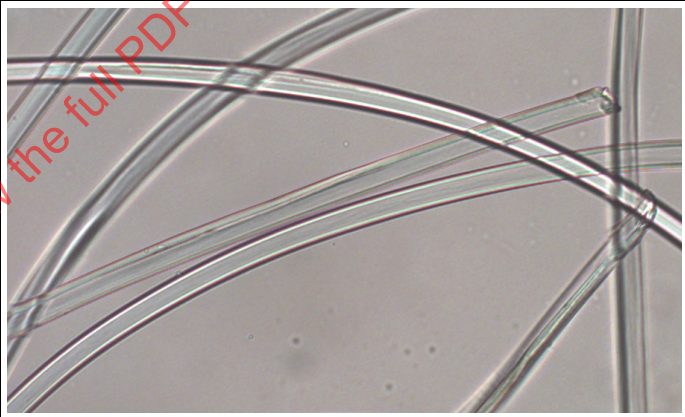


Figure B.26 — Light Microscopy longitudinal view of polyethylene / polypropylene bicomponent fibre

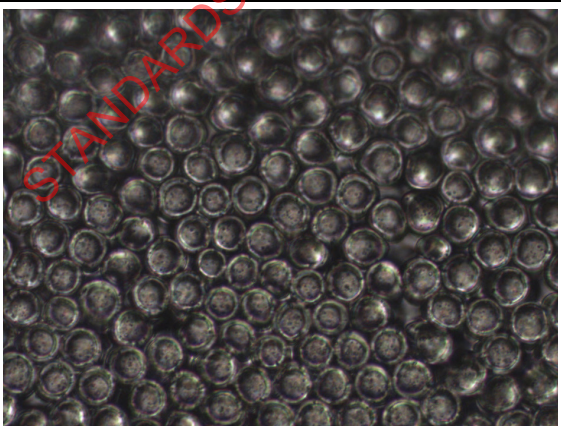


Figure B.27 — Light Microscopy - cross view of polyester / polyester bicomponent fibre

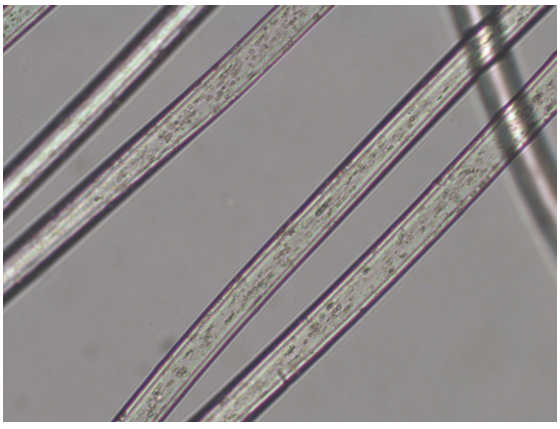


Figure B.28 — Light Microscopy longitudinal view of polyester / polyester bicomponent fibre

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Annex C
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Scanning Electron Micrographs of Fibres

C.1 Monocomponent fibres

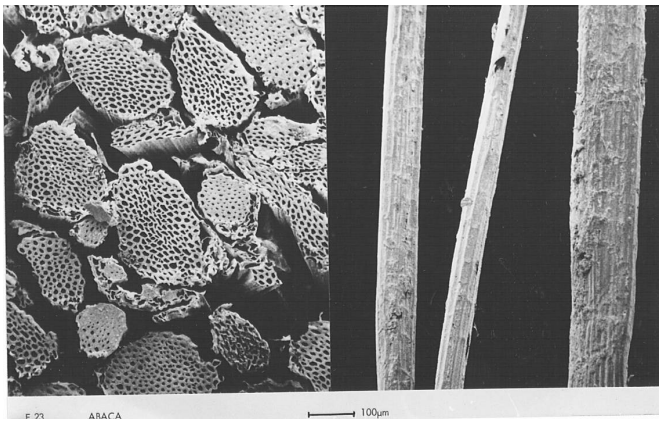


Figure C.1 — Abaca: cross and longitudinal view

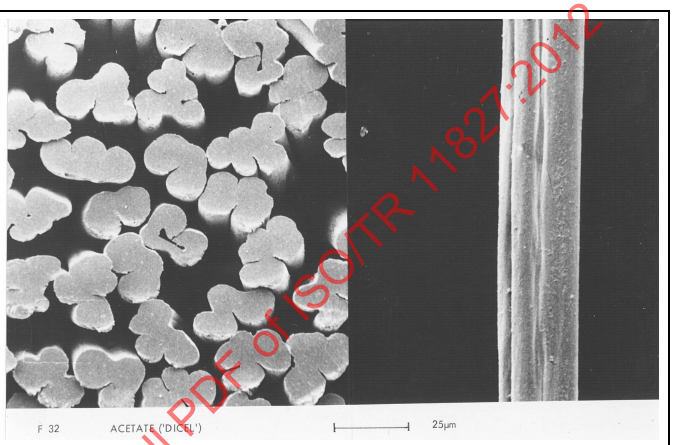


Figure C.2 — Acetate: cross and longitudinal view



Figure C.3 — Acrylic: cross and longitudinal view

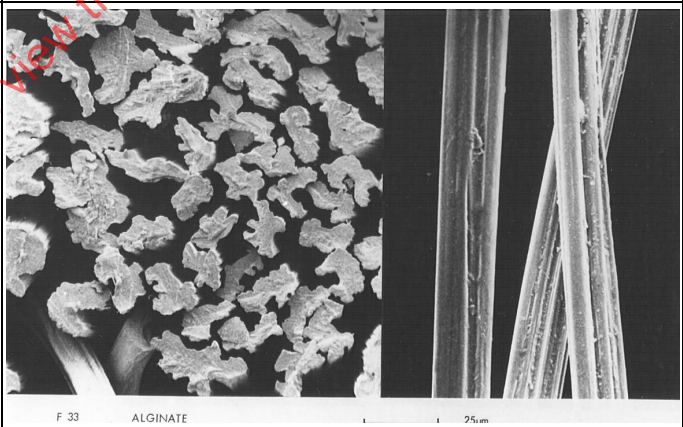


Figure C.4 — Alginate: cross and longitudinal view

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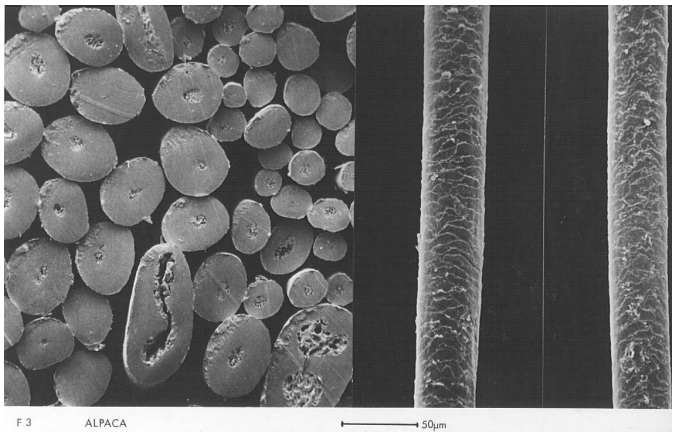


Figure C.5 — Alpaca: cross and longitudinal view

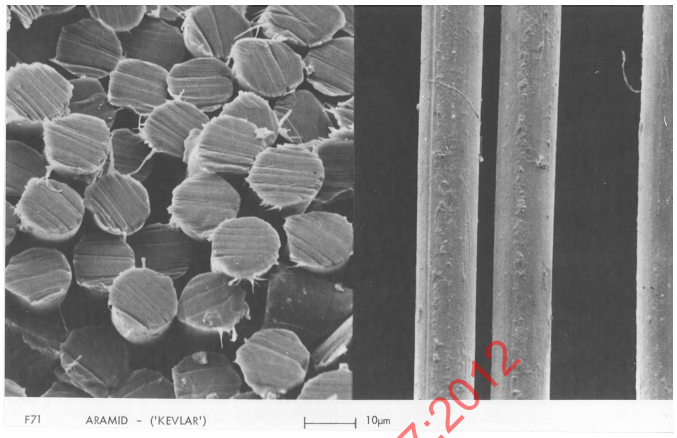


Figure C.6 — Aramid (para-): cross and longitudinal view

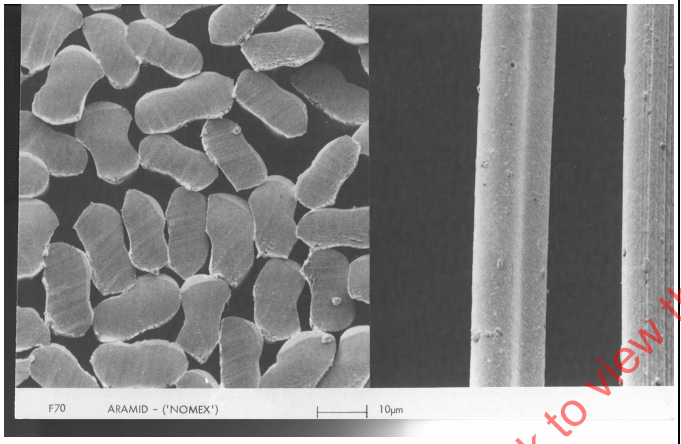


Figure C.7 — Aramid (meta-): cross and longitudinal view

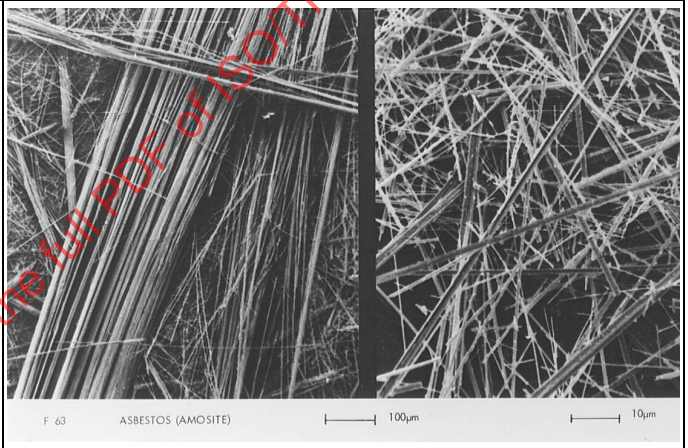


Figure C.8 — Asbestos (amosite): cross and longitudinal view

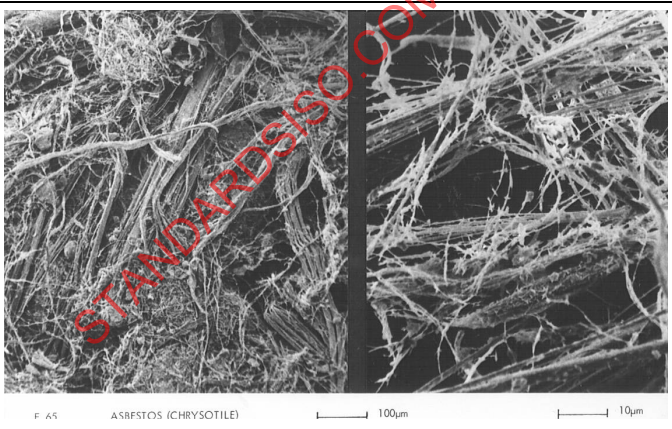


Figure C.9 — Asbestos (chrysotile): cross and longitudinal view

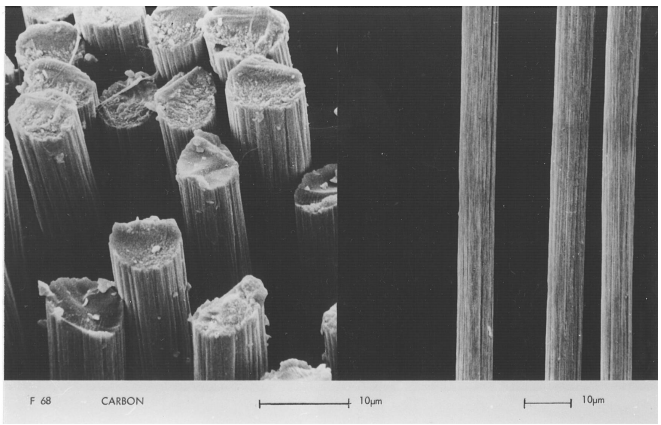


Figure C.10 — Carbon: cross and longitudinal view

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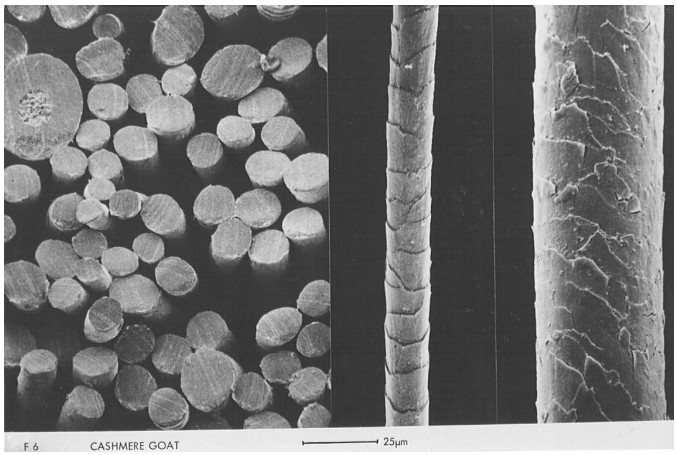


Figure C.11 — Cashmere: cross and longitudinal view

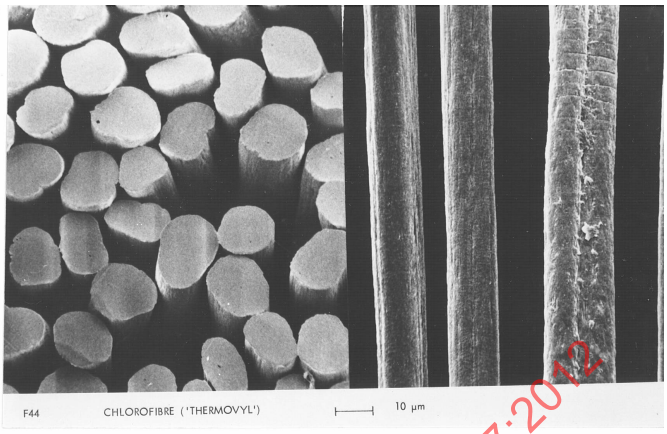


Figure C.12 — Chlorofibre: cross and longitudinal view

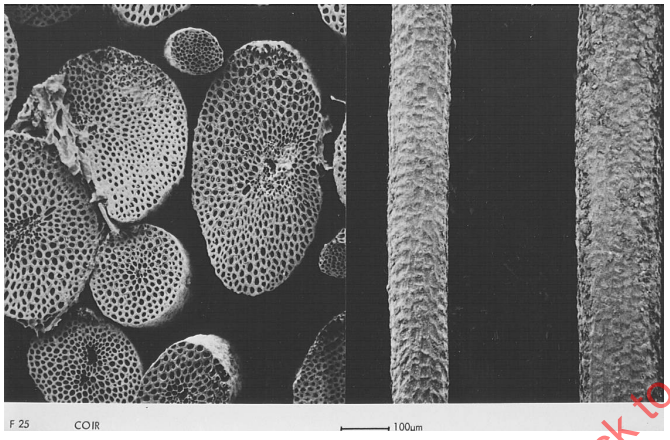


Figure C.13 — Coir: cross and longitudinal view



Figure C.14 — Cotton: cross and longitudinal view



Figure C.15 — Cupro: cross and longitudinal view

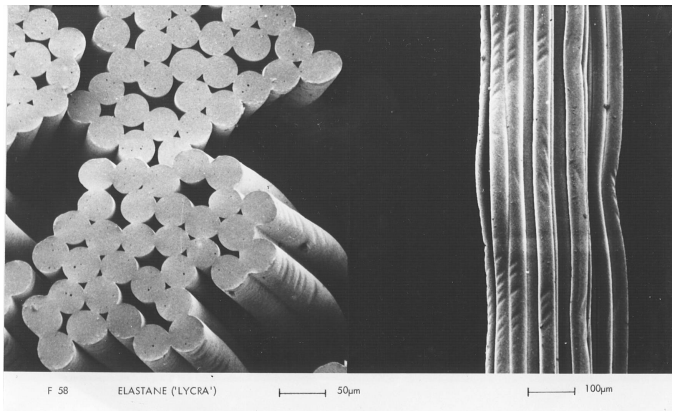


Figure C.16 — Elastane: cross and longitudinal view

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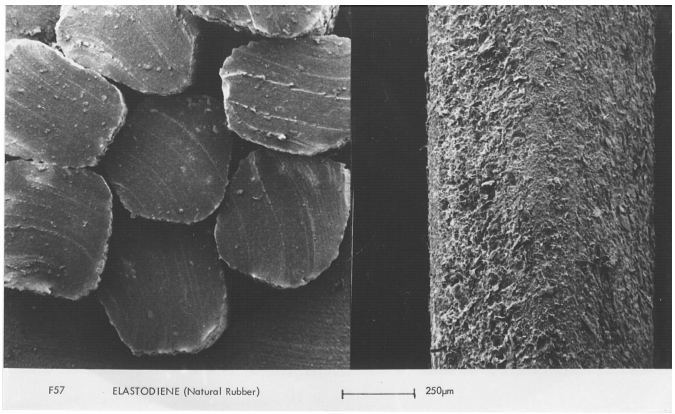


Figure C.17 — Elastodiene: cross and longitudinal view

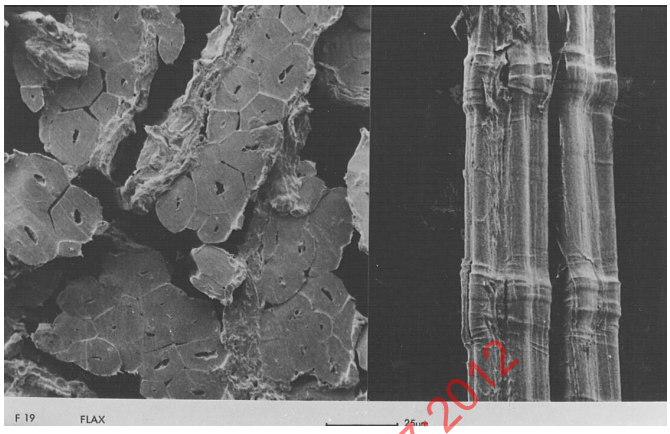


Figure C.18 — Linen (flax): cross and longitudinal view

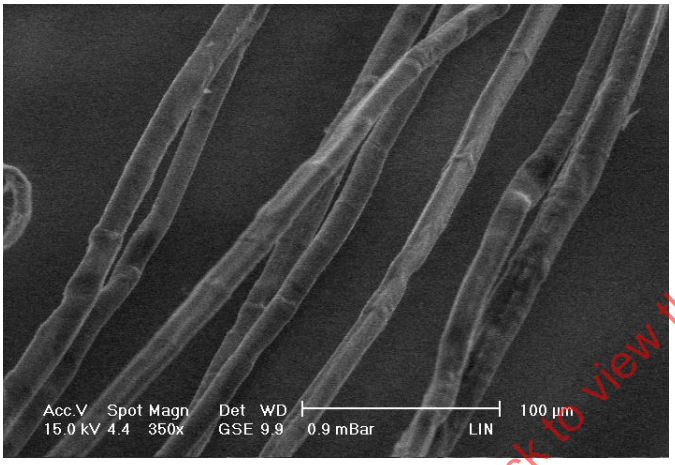


Figure C.19 — Flax: longitudinal view

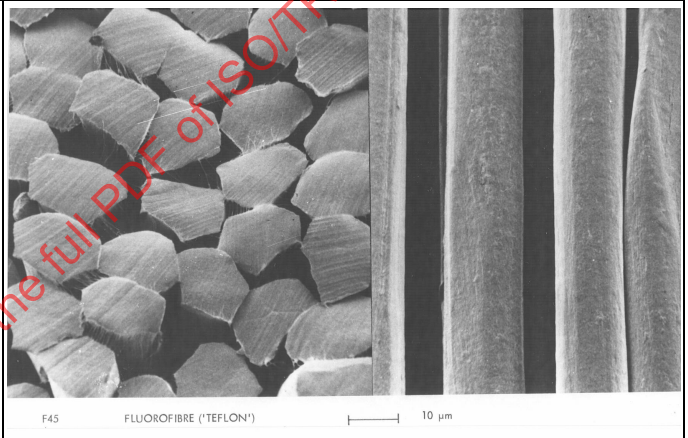


Figure C.20 — Fluorofibre: cross and longitudinal view



Figure C.21 — Glass: cross and longitudinal view

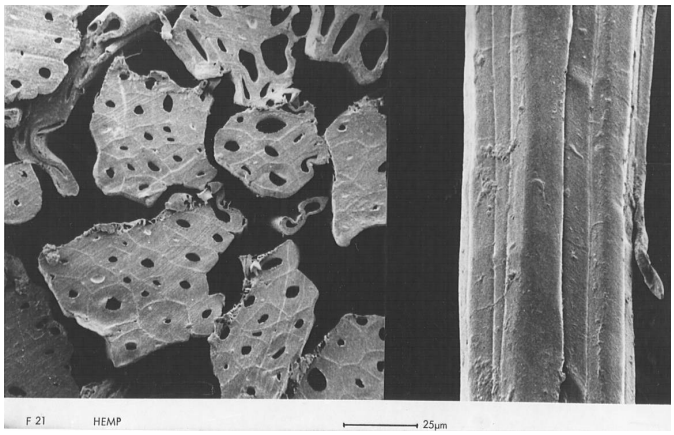


Figure C.22 — Hemp: cross and longitudinal view

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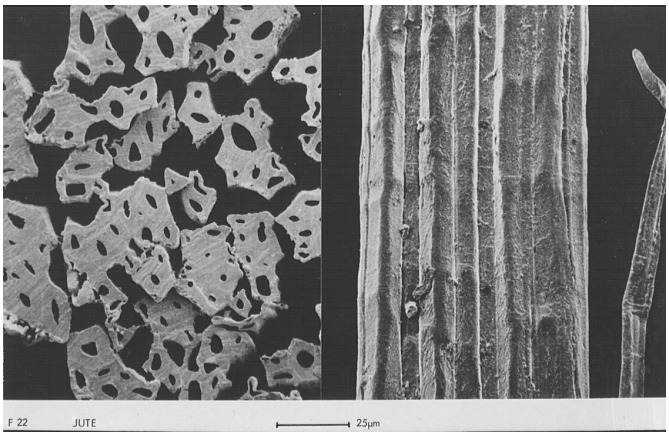


Figure C.23 — Jute: cross and longitudinal view

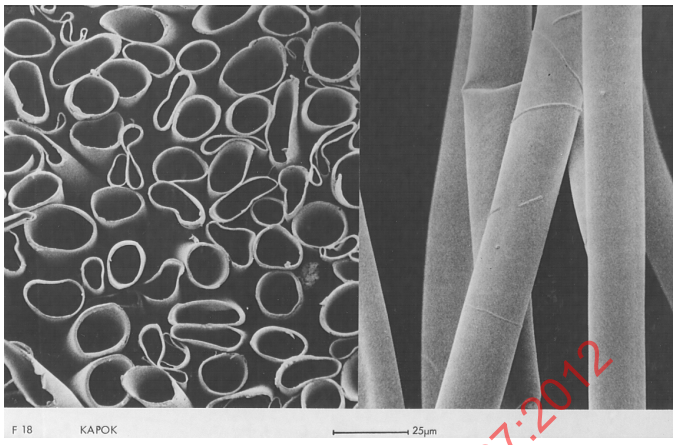


Figure C.24 — Kapok: cross and longitudinal view

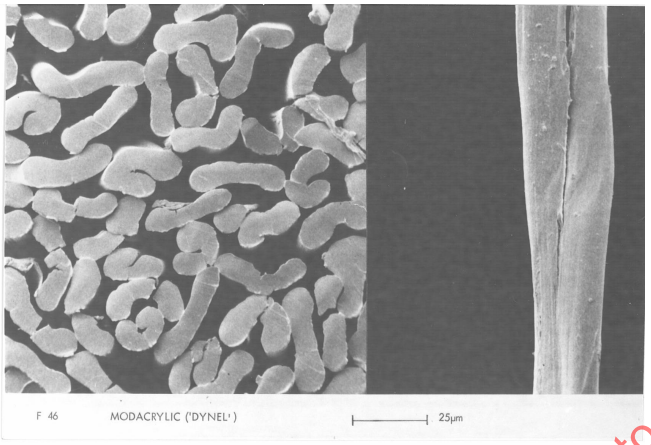


Figure C.25 — Modacrylic: cross and longitudinal view

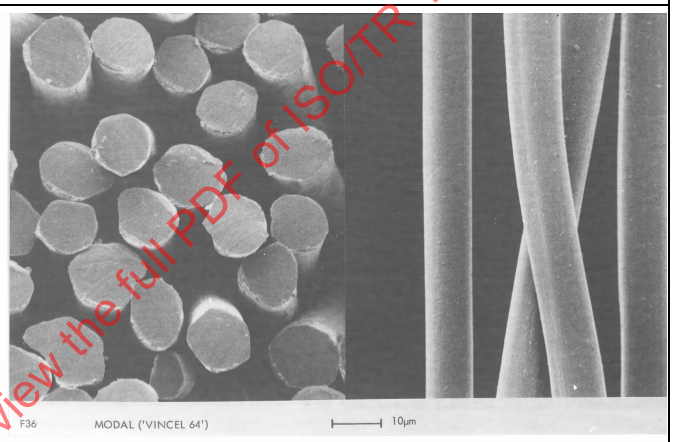


Figure C.26 — Modal: cross and longitudinal view



Figure C.27 — Polyamide: cross and longitudinal view

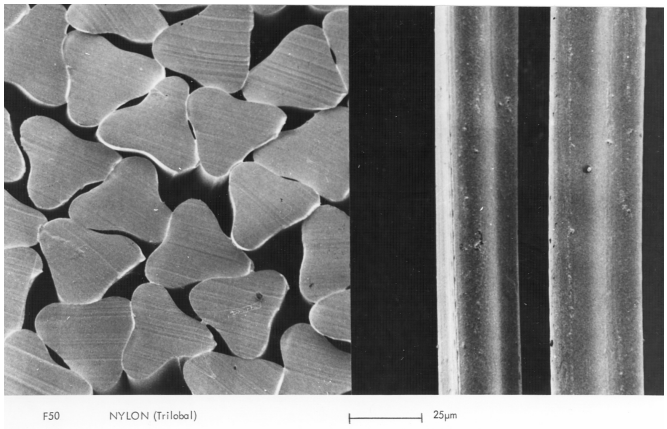


Figure C.28 — Polyamide (trilobal): cross and longitudinal view

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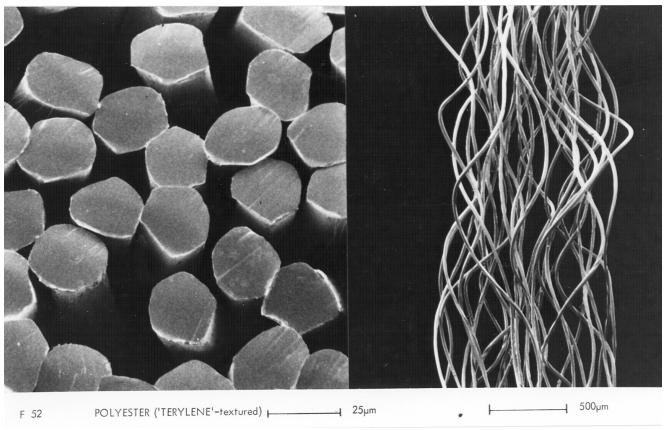


Figure C.29 — Polyester: cross and longitudinal view

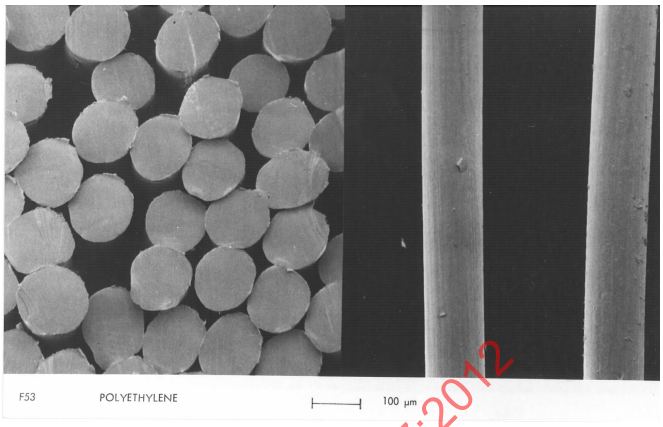


Figure C.30 — Polyethylene: cross and longitudinal view

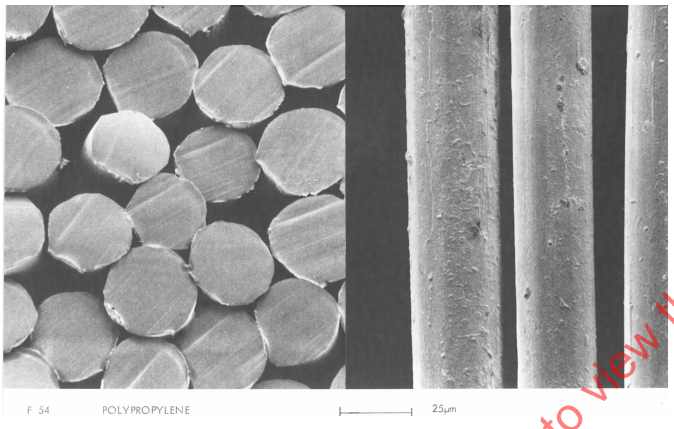


Figure C.31 — Polypropylene: cross and longitudinal view

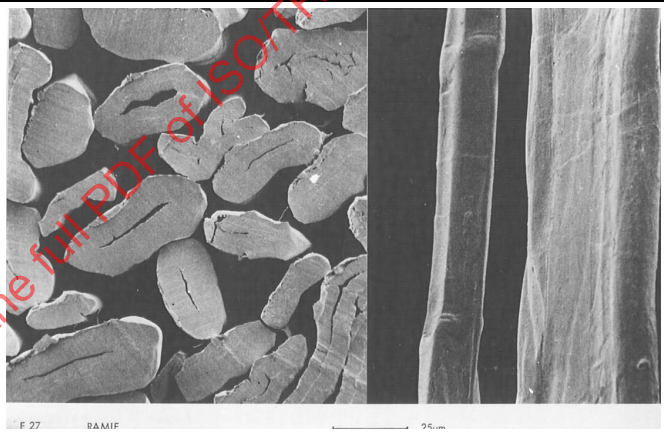


Figure C.32 — Ramie: cross and longitudinal view

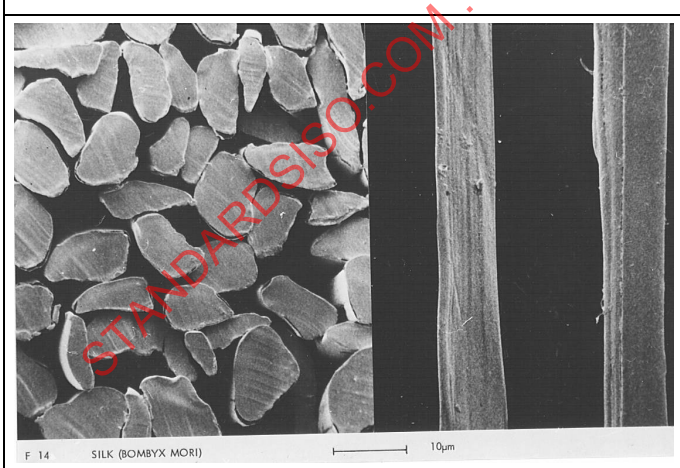


Figure C.33 — Silk (Bombyx Mori): cross and longitudinal view

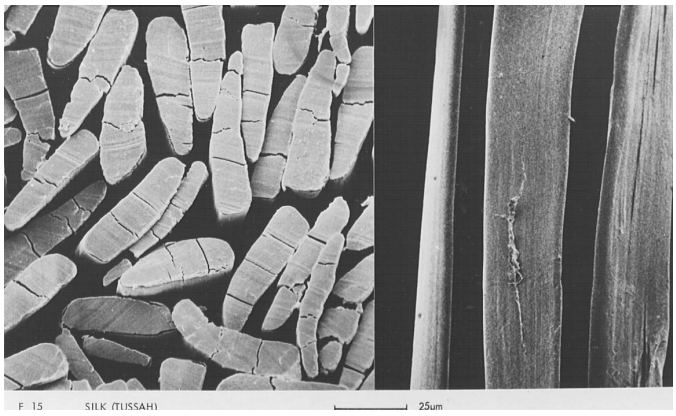


Figure C.34 — Silk (Tussah): cross and longitudinal view

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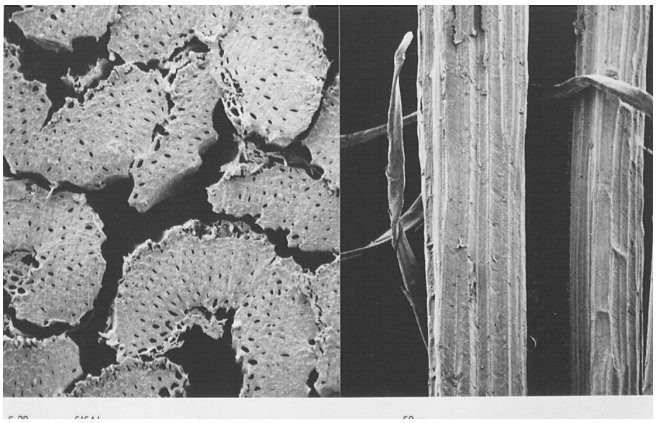


Figure C.35 — Sisal: cross and longitudinal view

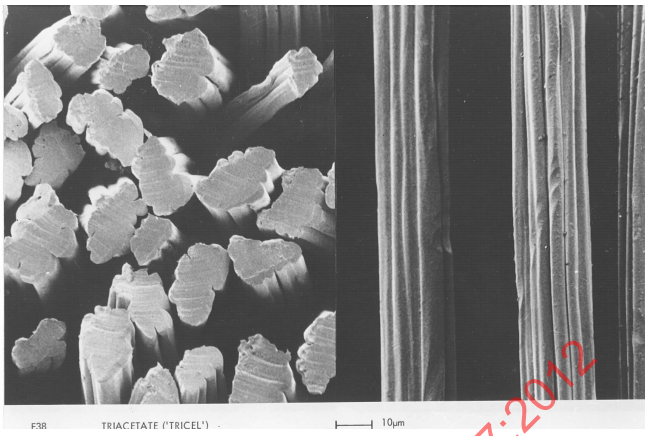


Figure C.36 — Triacetate: cross and longitudinal view

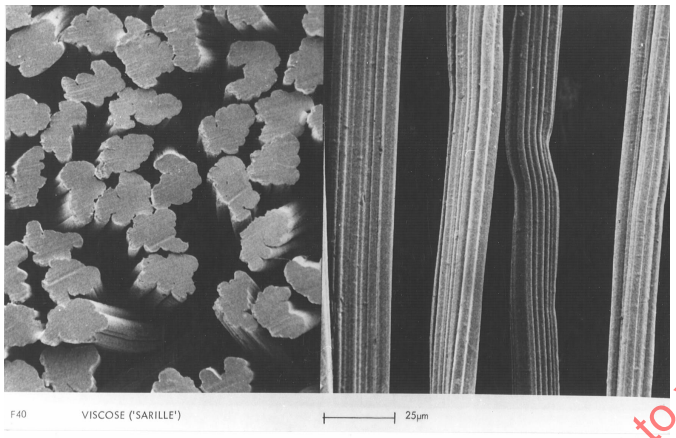


Figure C.37 — Viscose: cross and longitudinal view

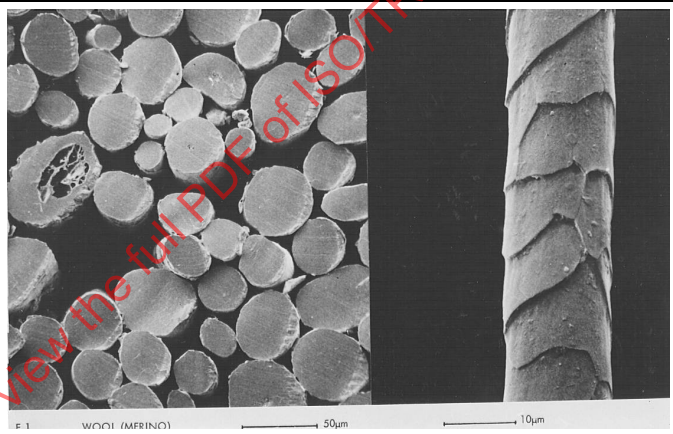


Figure C.38 — Wool: cross and longitudinal view



Figure C.39 — Wool: longitudinal view

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C.2 Bicomponent fibres

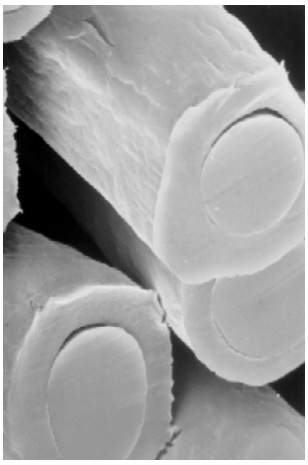


Figure C.40 — bicomponent fibres: cross view of core/sheath configuration

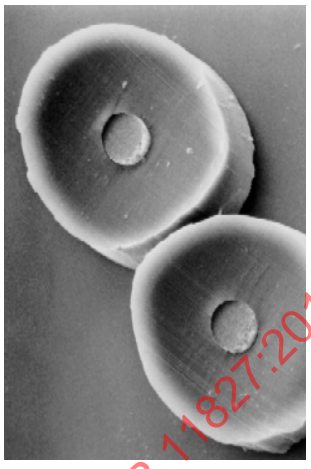


Figure C.41 — bicomponent fibres: cross view of core/sheath configuration

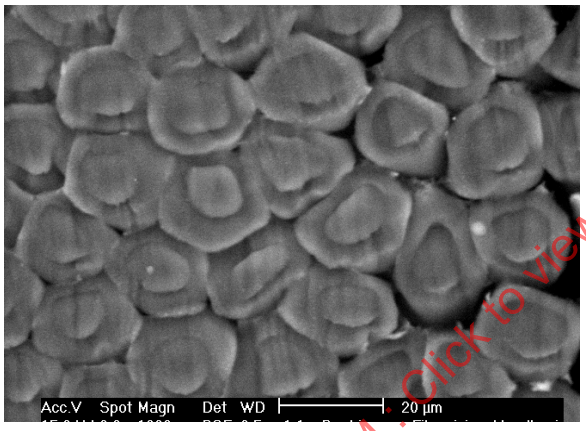


Figure C.42 — SEM: cross view of polyethylene / polypropylene bicomponent fibre

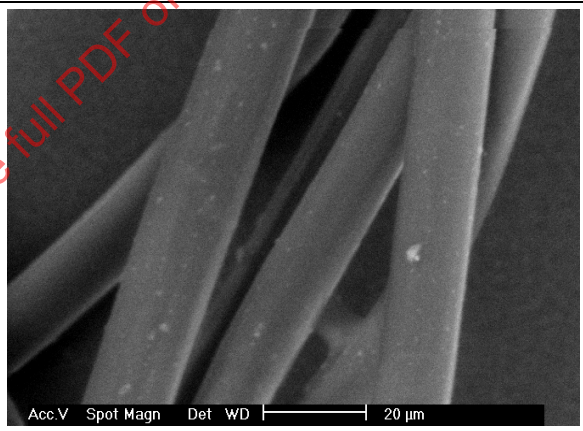


Figure C.43 — SEM: longitudinal view of polyethylene / polypropylene bicomponent fibre

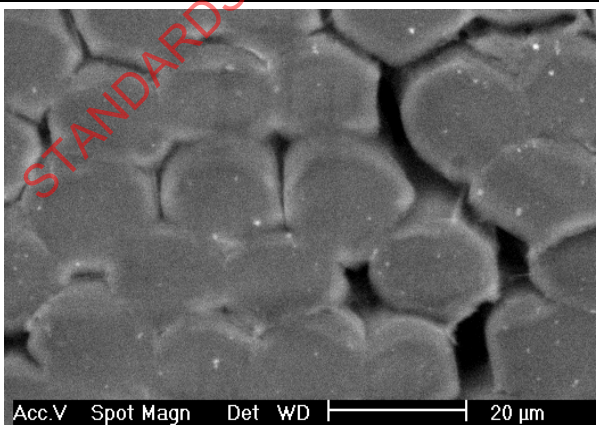


Figure C.44 — SEM: cross view of polyester / polyester bicomponent fibre

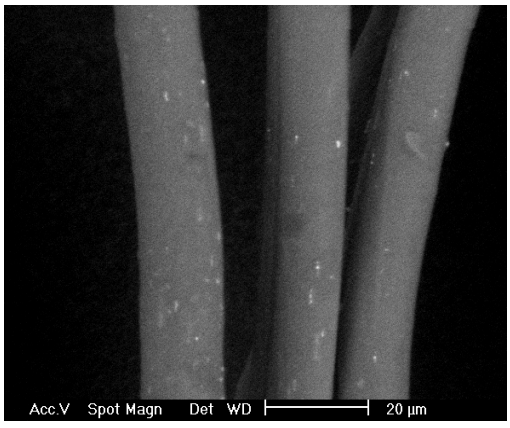


Figure C.45 — SEM: longitudinal view of polyester / polyester bicomponent fibre

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Annex D
(informative)

Solubility of fibres

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Table D.1 — Solubility of fibres

solvent	60 % Sulphuric acid		70 % Sulphuric acid		Concentrated Sulphuric acid		Concentrated nitric acid		Copper ethylene diamine		20 % Hydrochloric acid		35 % Hydrochloric acid		Glacial acetic acid		5 % Sodium hydroxide		Sodium hypochlorite		100 % Acetone	
Name of fibre	Normal temperature		Normal temperature		Normal temperature		Normal Temperature		Normal temperature		Normal temperature		Normal temperature				Boiling		Normal temperature			
Cotton	I		S	①	S	①	I		S	①	I		I		I	△	I		I		I	△
Hemp	I		S	①	S	①	I		S	②	I		I		I	△	I		I		I	△
Silk	S	①	S	①	S	①	I	swollen	S	○	SS	③	S	①	I	△	S	③	S	①	I	△
Wool	I		I		SS	③	I		I		I		I		I	△	S	②	S	②	I	△
Viscose, Cupro, Lyocell	CS ※	①	S	○	S	○	I		S	○	I		S	①	I	△	I		I		I	△
Acetate	S	①	S	○	S	○	S	○	I		I		S	○	S	□ ○	CS	③	I		S	□ ○
Triacetate	CS	③	S	②	S	①	S	①	I		I		SS	③	S	□ ①	I		I		S	□ ○
Polyamide 6	S	①	S	○	S	○	S	○	I		S	○	S	○	S	△ ○	I		I		I	△
Polyamide 6.6	S	①	S	○	S	○	S	○	I		S	○	S	○	S	△ ○	I		I		I	△
Vinylal	S	①	S	○	S	○	S	○	I		S	○	S	○	I	△	I		I		I	△
Acrylic	I		I		S	○	S	○	I		I		I		I	△	I		I		I	△
Modacrylic	I		I		S	○	CS	③	I		I		I		I	△	I		I		※ S	40～50 ℃ ○
Polyester	I		I		S	○	I		I		I		I		I	△	I		I		I	△
Polyvinyl chloride	I		I		I		I		I		I		I		I	△	I		I		SS	△ ③
Vinylidene	I		I		I		I		I		I		I		I	△	I		I		I	△
Polypropylene	I		I		I		I		I		I		I		I	△	I		I		I	△
elastane	※		※		※		※		I		I		※		※		I		I		I	△
Aramid (para-) [a]	I		I		S	○	I		I		I		I		I	△	I		I	[d]	I	△

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Table D.1 (*continued*)

solvent	60 % Sulphuric acid		70 % Sulphuric acid		Concentrated Sulphuric acid		Concentrated nitric acid		Copper ethylene diamine		20 % Hydrochloric acid		35 % Hydrochloric acid		Glacial acetic acid		5 % Sodium hydroxide		Sodium hypochlorite		100 % Acetone	
Name of fibre	Normal temperature		Normal temperature		Normal temperature		Normal Temperature		Normal temperature		Normal temperature		Normal temperature				Boiling		Normal temperature			
Aramid (para-) [b]	I		I		SS	③ Lump state	I		I		I		I		I	△	I		I		I	△
Aramid (meta-)	I		I		S	①	I		I		I		I		I	△	I		I		I	△
Polylactide	I		I		S	○	SS	③ Lump State	I		I		I		I	△	S	※	I		I	△

[a] this aramid type is made of one diamine and a chloride. This aramid type is broken up into fine segments by sodium hypochlorite

[b] this aramid type is made of two different diamines and a chloride.

I: Insoluble for 3 minute immersion / SS: Slightly Soluble / CS: Considerably Soluble / S: Soluble

□: Ambient room temperature / △: Boiling

○: at once, immediately / ①, ②, ③: for, respect., 1, 2 or 3 minutes

※: Solubility differs from types

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Annex E
(informative)

Examples of Infrared Spectra

E.1 Examples of spectra in relation to "% transmittance"

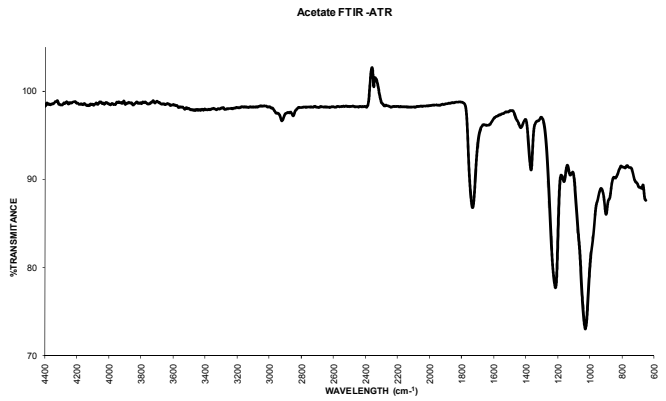


Figure E.1 — FT-IR spectra of acetate

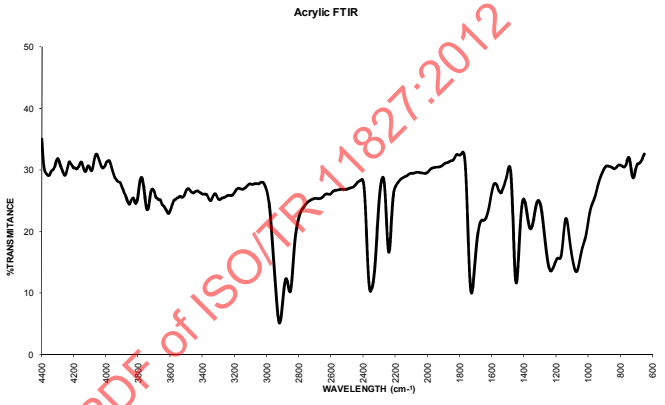


Figure E.2 — FT-IR spectra of acrylic

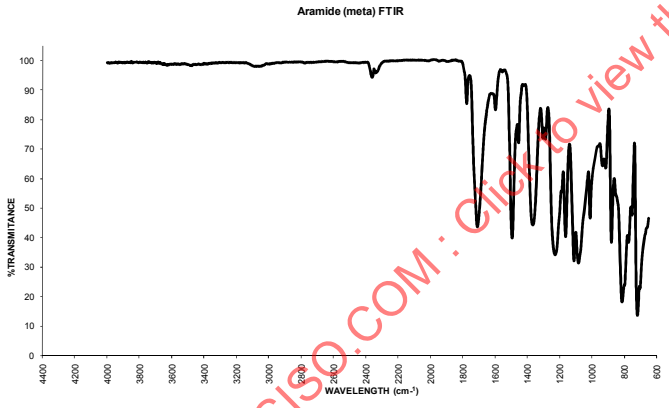


Figure E.3 — FT-IR spectra of meta-aramid

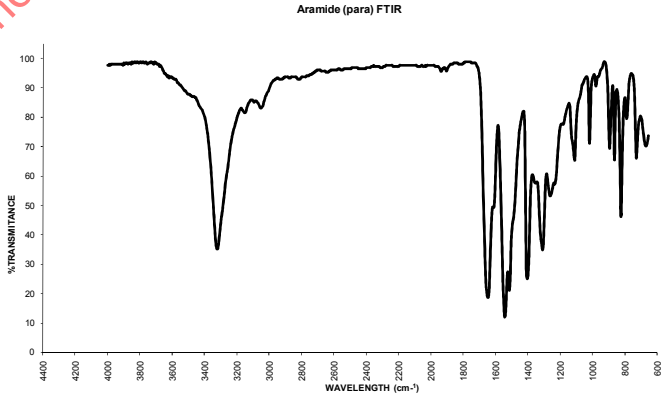


Figure E.4 — FT-IR spectra of para-aramid

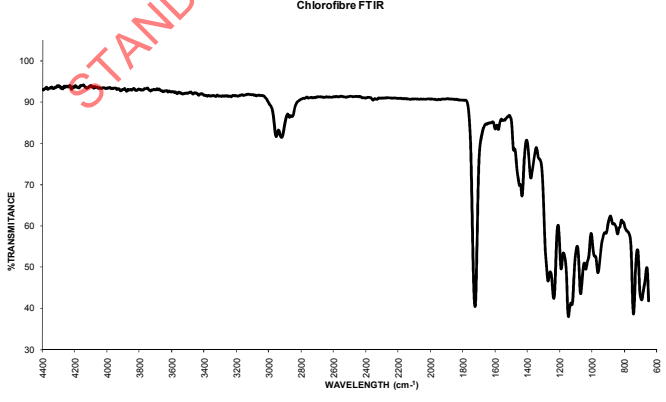


Figure E.5 — FT-IR spectra of chlorofibre

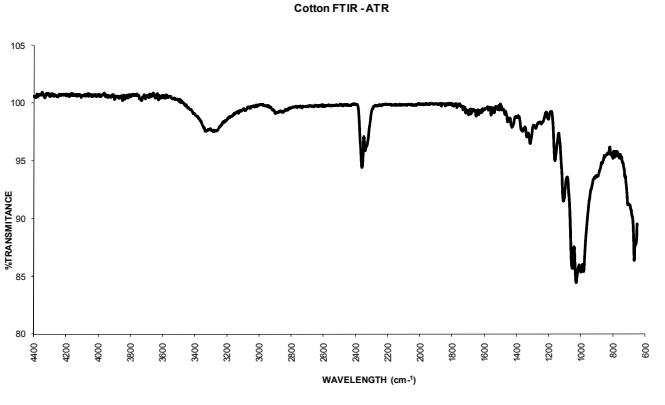


Figure E.6 — FT-IR spectra of cotton

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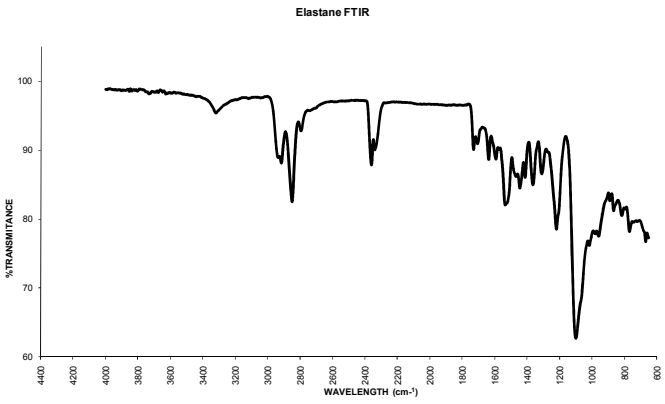


Figure E.7 — FT-IR spectra of elastane

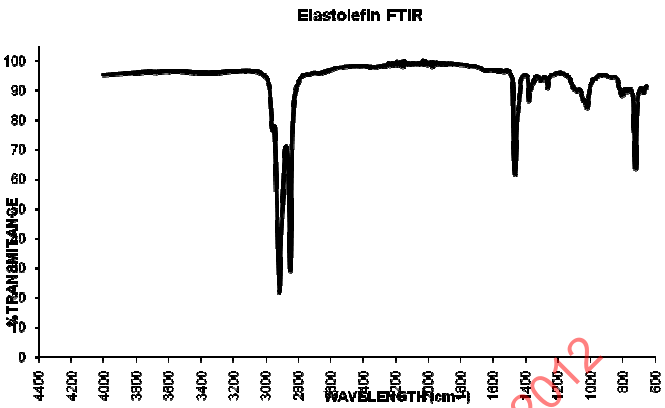


Figure E.8 — FT-IR spectra of elastolefin

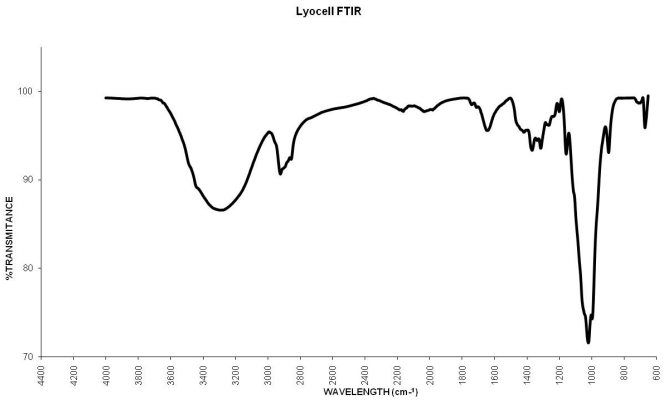


Figure E.9 — FT-IR spectra of lyocell

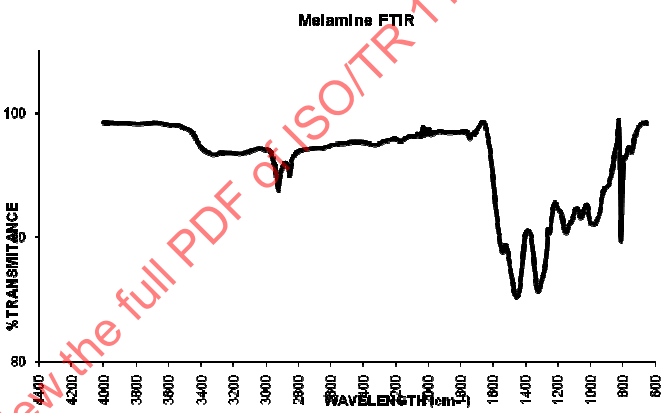


Figure E.10 — FT-IR spectra of melamine

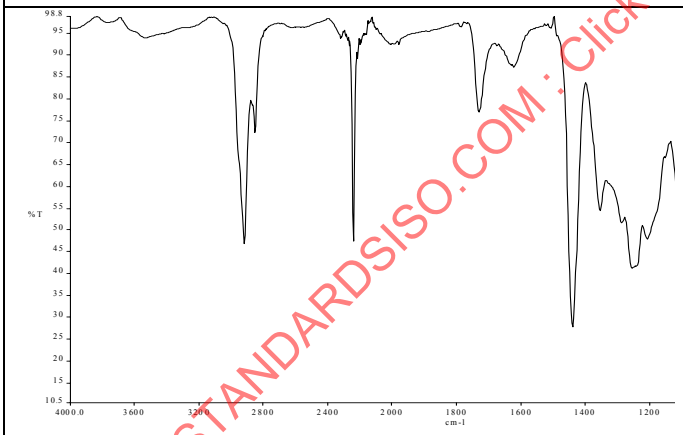


Figure E.11 — FT-IR spectra of modacrylic

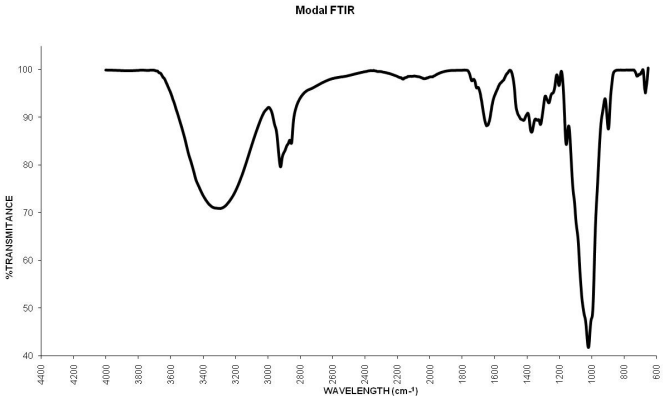


Figure E.12 — FT-IR spectra of modal

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