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**Textiles — Composition testing —  
Identification of fibres**

*Textiles — Essai de composition — Identification des fibres*

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Published in Switzerland

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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is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.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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# 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.

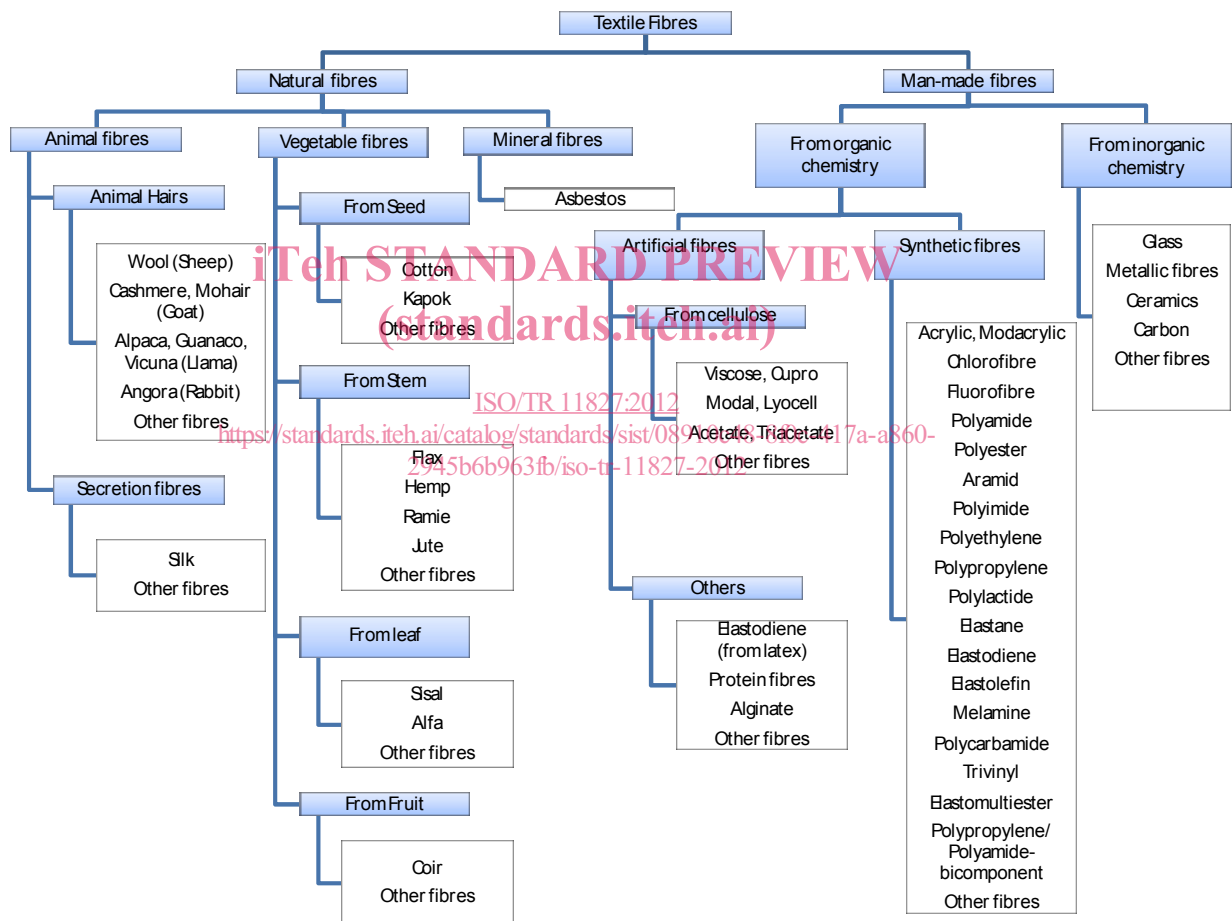


Figure 1 — Classification of the textile fibres in relation to their origin

## 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.

### 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

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

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**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



- 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.

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 ( $\rho$  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.

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**7 Techniques**

**7.1 Microscopy**

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**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.

### 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-bromonaphthalene 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 than 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.

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

Fibre		Refractive Index		Birefringence	
		Long $n_{//}$	Cross $n_{\perp}$	$\Delta n$	
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

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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 <sup>a</sup>	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
<sup>a</sup> refractive index of cedarwood oil changes with time	

## 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

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).

#### 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  $\mu\text{m}$ , that is to say from 4000  $\text{cm}^{-1}$  to 670  $\text{cm}^{-1}$  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.

### 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  $\mu\text{m}$  and 6,1  $\mu\text{m}$  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.