PD CEN ISO/TR 11827:2016



BSI Standards Publication

Textiles — Composition testing — Identification of fibres (ISO/TR 11827:2012)

Documents
Document Sharing Hub

Full document details: PD CEN ISO/TR 11827:2016

National foreword

This Published Document is the UK implementation of CEN ISO/TR 11827:2016. It is identical to ISO/TR 11827:2012. It supersedes PD ISO/TR 11827:2012 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee TCI/100, Co-ordination of activities in textiles and clothing.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2016.
Published by BSI Standards Limited 2016

ISBN 978 0 580 93121 5

ICS 59.060.01

Compliance with a British Standard cannot confer immunity from legal obligations.

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 30 June 2012.

Amendments/corrigenda issued since publication

Date	Text affected
30 June 2016	This corrigendum renumbers PD ISO/TR 11827:2012 as PD CEN ISO/TR 11827:2016

Documents
Document Sharing Hub

Full document details: PD CEN ISO/TR 11827:2016

TECHNICAL REPORT

RAPPORT TECHNIQUE

TECHNISCHER BERICHT

CEN ISO/TR 11827

June 2016

ICS 59.060.01

English Version

Textiles - Composition testing - Identification of fibres (ISO/TR 11827:2012)

Textiles - Essai de composition - Identification des fibres (ISO/TR 11827:2012)

This Technical Report was approved by CEN on 22 May 2016. It has been drawn up by the Technical Committee CEN/TC 248.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

© 2016 CEN All rights of exploitation in any form and by any means reserved worldwide for CEN national Members.

Ref. No. CEN ISO/TR 11827:2016 E

Documents
Document Sharing Hub

Full document details: PD CEN ISO/TR 11827:2016

European foreword

This document (CEN ISO/TR 11827:2016) has been prepared by Technical Committee ISO/TC 38 “Textiles” in collaboration with Technical Committee CEN/TC 248 “Textiles and textile products” the secretariat of which is held by BSI.

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

Endorsement notice

The text of ISO/TR 11827:2012 has been approved by CEN as CEN ISO/TR 11827:2016 without any modification.

Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

Contents

Page

Foreword iv

Introduction v

1 Scope 1

2 Safety note 1

3 Normative references 2

4 Terms and definitions 2

5 Principle 2

6 Apparatus and preparation of solutions 3
6.1 Apparatus 3
6.2 Preparation of solutions 3

7 Techniques 4
7.1 Microscopy 4
7.2 Flame tests 6
7.3 Staining Tests 7
7.4 Solubility Tests 7
7.5 Infrared Spectroscopy 8
7.6 Thermal Analysis 12
7.7 Density measurement methods 14
7.8 Other Instrumental Methods 14

8 Examples of procedures 15
8.1 Procedure using microscopy, solubility tests and FT-IR tests (examples) 15
8.2 Procedure using solubility tests (examples) 17
8.3 Procedure using combustion tests and melting point determination (example) 19
8.4 Procedure using microscopy, FT-IR analysis and thermal analysis, case of bicomponent fibres (examples) 19

Annex A (informative) **Characteristics relative to fibre identification testing** 24

Annex B (informative) **Photomicrographs of Fibres (Light Microscopy)** 29

Annex C (informative) **Scanning Electron Micrographs of Fibres** 34

Annex D (informative) **Solubility of fibres** 42

Annex E (informative) **Examples of Infrared Spectra** 45

Annex F (informative) **Thermal transition temperature** 50

Annex G (informative) **Density** 54

Annex H (informative) **Alphabetical index of figures** 55

Bibliography 57

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

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.*

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

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.

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

Documents
Document Sharing Hub

This page deliberately left blank

Full document details: PD CEN ISO/TR 11827:2016

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.

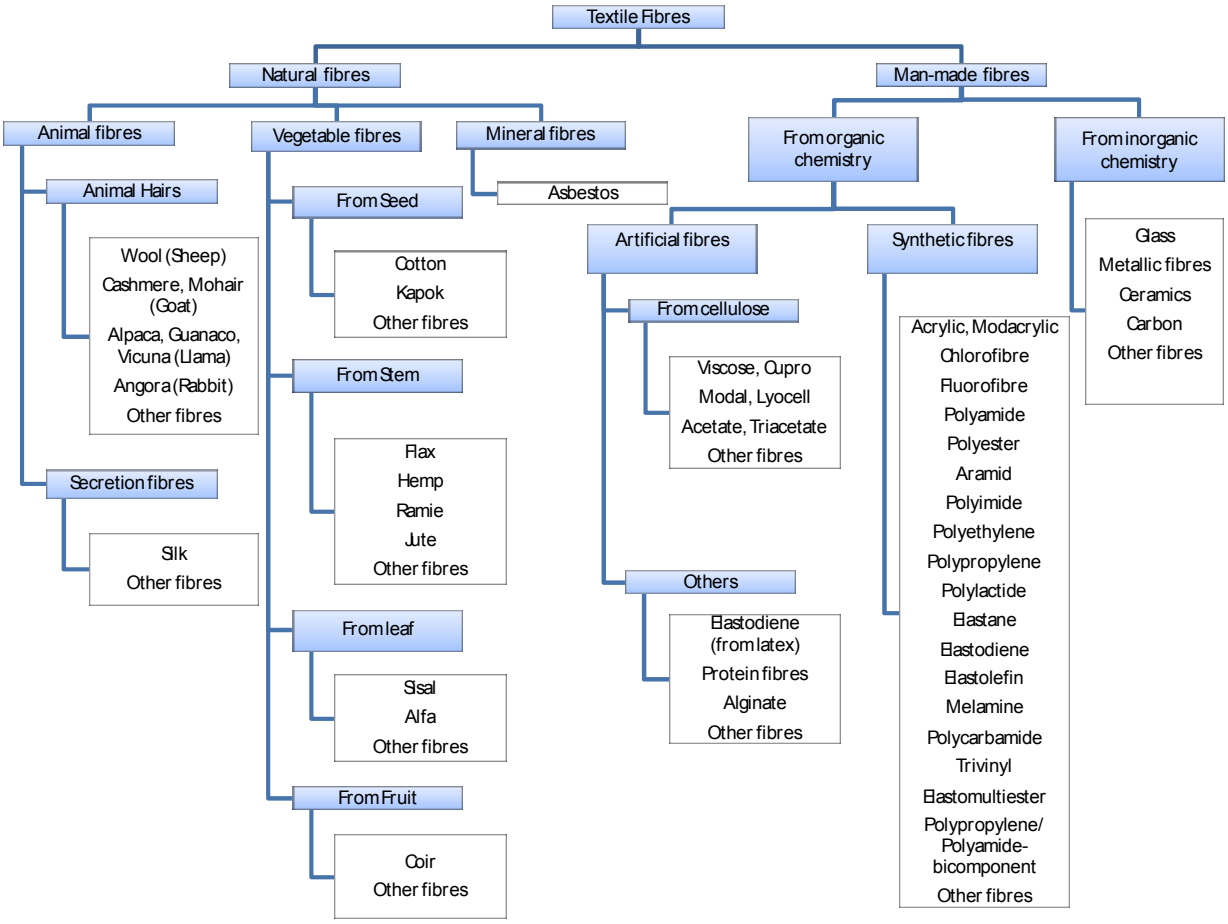


Figure 1 — Classification of the textile fibres in relation to their origin

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.

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

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

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

- 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.

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

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.

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

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.

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

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	

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

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

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).

Documents
Document Sharing Hub

© ISO 2012 – All rights reserved

Full document details: PD CEN ISO/TR 11827:2016

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.

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

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.

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

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.

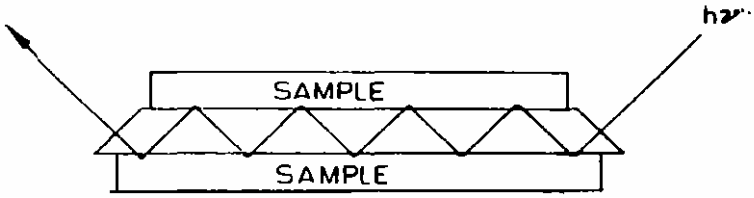


Figure 2 — Multiple internal effect

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.

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

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

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

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.

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016

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 %.

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: PD CEN ISO/TR 11827:2016