BS ISO 4650:2012

BSI Standards Publication

Rubber — Identification — Infrared spectrometric methods

bsi.

...making excellence a habit.™

Document Sharing Hub

Full document details: BS ISO 4650:2012

National foreword

This British Standard is the UK implementation of ISO 4650:2012. It supersedes BS ISO 4650:2005 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/23, Test methods for rubber and non-black compounding ingredients.

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 2013. Published by BSI Standards Limited 2013

ISBN 978 0 580 65979 9

ICS 83.060

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

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 January 2013.

Amendments issued since publication

Date	Text affected

Documents
Document Sharing Hub

Full document details: BS ISO 4650:2012

INTERNATIONAL STANDARD

ISO 4650

Third edition
2012-12-15

Rubber — Identification — Infrared spectrometric methods

Caoutchouc — Identification — Méthodes spectrométriques dans l'infrarouge

Reference number
ISO 4650:2012(E)

Documents
Document Sharing Hub

© ISO 2012

Full document details: BS ISO 4650:2012

COPYRIGHT PROTECTED DOCUMENT

© ISO 2012

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub

Full document details: BS ISO 4650:2012

Contents

Page

Foreword ... **iv**

1 Scope ... **1**

2 Normative references ... **1**

3 Principle ... **1**
3.1 Analysis by transmission ... 1
3.2 Analysis by reflectance ... 1

4 Types of rubber ... **2**
4.1 General ... 2
4.2 Exceptions for blends ... 3
4.3 Reference spectra ... 4

5 Reagents ... **4**

6 Apparatus ... **4**

7 Procedure for infrared spectrometric examination using the transmission technique ... **6**
7.1 Procedure for raw rubber films moulded or cast from solution ... 6
7.2 Procedure for raw rubbers, vulcanizates and films obtained from pyrolysate ... 6
7.3 Procedure for vulcanized rubber film obtained after evaporation of the solution solvent ... 7

8 Procedure for attenuated total reflectance (ATR or micro-ATR) surface analysis ... **8**

9 Interpretation of spectra ... **8**
9.1 Reference spectra ... 8
9.2 Tables of diagnostic absorptions ... 9

10 Test report ... **9**

Annex A (informative) **Comparison of reflectance and transmission spectra** ... **10**

Annex B (informative) **Absorption characteristics and reference spectra** ... **11**

Bibliography ... **88**

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012

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.

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 4650 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This third edition cancels and replaces the second edition (ISO 4650:2005), which has been technically revised.

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012

INTERNATIONAL STANDARD ISO 4650:2012(E)

Rubber — Identification — Infrared spectrometric methods

1 Scope

This International Standard specifies two methods for the identification of rubbers, including thermoplastic elastomers, either in the raw state or in the form of vulcanized or unvulcanized mixes. The first method is based on infrared spectrometric examination using the transmission technique. The second method makes use of analysis by reflectance.

A comparison of the spectra resulting from reflectance (attenuated total reflectance ATR) and transmission (film) is given in Annex A.

Both methods comprise examination of polymers by their pyrolysis products (pyrolysates), or by films cast from solution or obtained by moulding (for raw rubbers only).

Typical spectra are given in Annex B.

The principle of the methods implies that sample preparation and analysis of the infrared spectra are carried out by experienced personnel and that the equipment used for the production of spectra is operated in accordance with the manufacturer's instructions for optimum performance. Details of the operation of infrared spectrometers are not included in this International Standard.

The methods specified are qualitative methods only.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable to its application. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1407, *Rubber — Determination of solvent extract*

ISO 18064, *Thermoplastic elastomers — Nomenclature and abbreviated terms*

3 Principle

3.1 Analysis by transmission

The extractable material is first extracted from a test sample of the rubber and the rubber then prepared under precise conditions for spectroscopy in the form of raw polymer film, vulcanizate pyrolysate or vulcanizate film. The IR spectrum is recorded and then interpreted by comparison with a set of typical reference spectra.

3.2 Analysis by reflectance

This technique is directly applicable to raw rubbers and to vulcanized and unvulcanized rubbers. In the case of vulcanized and unvulcanized rubbers, the presence of organic or mineral additives can affect the interpretation of the spectrum.

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012

4 Types of rubber

4.1 General

Both methods are applicable to rubbers in the raw state and, if compounded, in both the vulcanized and unvulcanized states. They are applicable to the following types of rubber occurring either alone or in a binary mixture when the proportion of the minor component is, in general, between 10 % to 20 % by mass of the mixture (see, however, exceptions in 4.2).

4.1.1 M group

4.1.1.1 Acrylic rubber (ACM): Copolymer of ethyl acrylate (or other acrylates) and a small amount of a monomer which facilitates vulcanization.

4.1.1.2 Copolymer of ethyl acrylate (or other acrylates) and ethylene (AEM).

4.1.1.3 Chloropolyethylene (CM) and chlorosulfonylpolyethylene (CSM): The methods will not differentiate CM from CSM, and it will not differentiate between different types of CSM.

4.1.1.4 Ethylene-propylene copolymer (EPM) and ethylene-propylene-diene terpolymer (EPDM): The methods will not differentiate between the two types of polymer. However, examination of the spectrum gives some information about the ethylene-to-propylene ratio.

4.1.1.5 Fluorocarbon rubber (FKM): Examination of the pyrolysate may give some information about the different grades of fluorocarbon rubber present.

4.1.2 O group

4.1.2.1 Polychloromethyloxirane (CO): Copolymer of ethylene oxide and chloromethyloxirane (ECO) and terpolymers (GECO). Examination of the pyrolysate will not differentiate between different types of CO.

4.1.3 Q group

4.1.3.1 Polydimethylsiloxane (MQ), polymethylphenylsiloxane (PMQ) and polymethylfluorosiloxane (FMQ): Examination of the pyrolysate will differentiate PMQ from MQ.

4.1.4 R group

4.1.4.1 Butadiene rubber (BR): Examination of the pyrolysate will not differentiate between butadiene rubbers having different isomer ratios. However, examination of a raw rubber film gives some information about the isomer ratio.

4.1.4.2 Chloroprene rubber (CR): The methods will not differentiate between the different types of CR.

4.1.4.3 Isobutene-isoprene rubber (IIR) and halogenated isobutene-isoprene rubbers (BIIR and CIIR) and brominated isobutylene-co-*p*-methylstyrene (BIMS): Under the conditions used, it is not possible to differentiate between IIR, BIIR, CIIR and polyisobutene.

4.1.4.4 Natural rubber (NR) and synthetic isoprene rubber (IR): Natural rubber (1,4-*cis*-polyisoprene), gutta percha, balata (1,4-*trans*-polyisoprene) and synthetic isoprene rubber, whatever their microstructure (1,4-*cis*, 1,4-*trans* or 3,4-), are included.

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012

4.1.4.4.1 Examination of a rubber film will differentiate between 1,4-*cis*, 1,4-*trans* and 3,4-polyisoprenes; for non-extracted rubbers, it will differentiate natural rubber from 1,4-*cis* synthetic isoprene rubber, and 1,4-*trans* natural polyisoprenes from their synthetic counterparts. Examination of the pyrolysate film obtained from a vulcanizate provides no information on the microstructure of the polyisoprene or its origin, whether natural or synthetic.

4.1.4.5 Acrylonitrile-butadiene rubber (NBR): The methods will differentiate carboxylic acrylonitrile-butadiene rubbers (XNBRs) from hydrogenated acrylonitrile-butadiene rubbers (HNBRs). Associations of butadiene copolymers and PVC are included. Examination of the pyrolysate film gives some information about the acrylonitrile content.

4.1.4.6 Styrene-butadiene rubber (SBR): The methods will differentiate α-methylstyrene-butadiene rubbers from styrene-butadiene rubbers. Copolymers of styrene and butadiene, as well as of their substituted derivatives (e.g. α-methylstyrene), are included. Examination of a pyrolysate will not differentiate emulsion-polymerized rubbers from solution-polymerized rubbers. However, examination of a spectrum gives some information about the monomer ratio.

4.1.4.7 Polynorbornene.

4.1.5 T group

4.1.5.1 Polysulfide rubbers.

4.1.6 U group

4.1.6.1 Polyester urethane (AU) and polyether urethane (EU): The methods cover only millable polyurethanes.

4.1.7 TPE group

4.1.7.1 As defined in ISO 18064.

4.2 Exceptions for blends

4.2.1 Analysis of a blend of ethylene-propylene rubber with other rubbers presents difficulties when its ethylene-propylene content is below 40 %.

4.2.2 The methods will not differentiate between blends of ethylene-propylene rubber with chlorinated polyethylene and/or chloro-sulfonated polyethylene.

4.2.3 Analysis of a blend of natural and/or synthetic polyisoprene and chloroprene rubber may present difficulties, and identification of the minor component may only be possible when the content is equal to or greater than 30 % in the blend.

4.2.4 The methods will not differentiate NBR from NBR/BR blends or NBR blends, nor will it differentiate SBR from SBR/BR blends or SBR blends.

4.2.5 The presence of high quantities of sulfur in a vulcanizate may affect some characteristic bands.

4.2.6 The methods will not differentiate NBR/PVC blends from blends of NBR with other halogenated polymers or additives.

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012

4.3 Reference spectra

Tables of absorption characteristics and reference spectra from 4 000 cm^{-1} to 600 cm^{-1} for typical rubbers are given in Annex B.

5 Reagents

5.1 Nitrogen, in pressurized cylinders.

5.2 Extraction solvents, chosen to achieve maximum extraction (alternative solvents may be used on condition that it can be shown that they do not interfere with the interpretation of the infrared spectrum):

5.2.1 Methanol.

5.2.2 Acetone.

5.3 Solvents for rubber dissolution and film preparation, water-free and free from residues (see ISO 1407):

5.3.1 Chloroform.

5.3.2 1,2-dichlorobenzene.

5.4 Sodium sulfate, anhydrous.

5.5 Universal pH-indicator paper.

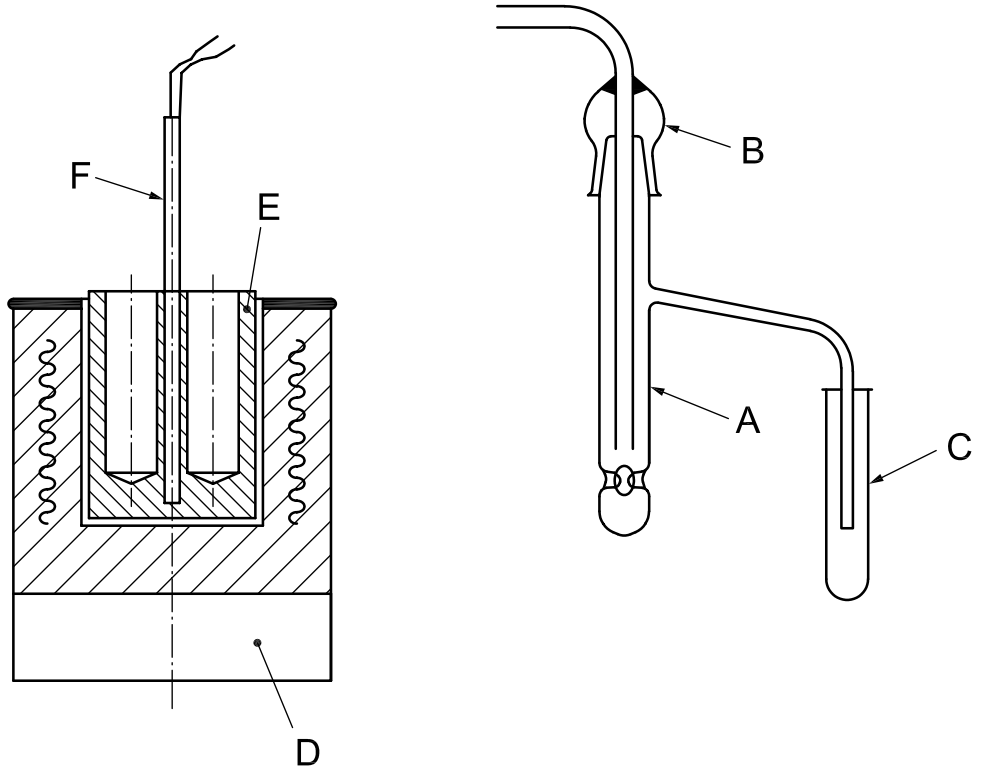
6 Apparatus

6.1 Extraction apparatus.

The apparatus specified in ISO 1407 is satisfactory.

6.2 Pyrolysis apparatus (see Figure 1), comprising a glass tube A having inward projections to prevent the sample from falling to the bottom of the tube, and a lateral condenser tube. The tube A has a standard ground-glass joint B that carries a small glass adductor tube. A collecting tube C is placed under the condenser tube. A thermoregulated electric furnace D accommodates an aluminium block E with holes for one or more tubes A.

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



Key

A glass tube for sample
B ground-glass joint
C collecting tube
D thermoregulated electric furnace
E aluminium block, bored to hold tubes
F thermocouple

Figure 1 — Temperature-controlled pyrolysis apparatus

6.3 Capillary pipettes.

6.4 Oven, capable of being maintained at 200 °C ± 5 °C.

6.5 Water bath.

6.6 Polished potassium bromide salt plates.

Other infrared transmission materials may be used if they show sufficient transmittance for analysis within a wavenumber range between 4 000 cm^{-1} and 600 cm^{-1}.

6.7 Filter aid, e.g. diatomaceous earth or similar.

6.8 Infrared spectrometer, of either the Fourier transform or dispersive type, with a wavenumber range of 4 000 cm^{-1} to 600 cm^{-1} and a spectral resolution of 4 cm^{-1} or higher.

6.9 ATR (attenuated total reflectance) attachment with a suitable reflecting crystal (e.g. diamond, ZnSe, Ge).

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012

7 Procedure for infrared spectrometric examination using the transmission technique

7.1 Procedure for raw rubber films moulded or cast from solution

7.1.1 Using a suitable solvent (see 5.2), extract the extractable material from a test sample of 2 g to 5 g in accordance with the procedure given in ISO 1407.

7.1.2 Dissolve a sufficient amount of the extracted rubber in a suitable solvent (see 5.3), at room temperature or under reflux, to give a concentrated solution.

7.1.3 Place a few drops of the concentrated solution on a potassium bromide salt plate (6.6) and allow the solvent to evaporate.

7.1.4 Films of raw rubber of a suitable thickness may also be obtained by moulding.

7.1.5 Record the spectrum from 4 000 cm^{-1} to 600 cm^{-1} using the infrared spectrometer (6.8).

7.1.6 After recording the spectrum, verify that no solvent absorption bands are present and check that the bands of the spectrum are neither off-scale nor too low. If these conditions are not met, repeat the preparation procedure on a fresh test sample and record a new spectrum.

7.1.7 A test for halogens may be carried out as described in 7.2.1.4.

7.2 Procedure for raw rubbers, vulcanizates and films obtained from pyrolysate

NOTE The methods described in 7.2.1 and 7.2.2 may give different relative absorbances for the polymers in a given blend.

7.2.1 Preferred method: Temperature-controlled pyrolysis in a stream of nitrogen

7.2.1.1 Extract a 2 g to 5 g test sample in accordance with the procedure given in ISO 1407.

7.2.1.2 Depending on the nature of the composition of the unknown vulcanizate and of the type of apparatus used, place 0,5 g to 2 g of the extracted, dried test sample in the pyrolysis tube A (see Figure 1).

7.2.1.3 Introduce a small quantity of sodium sulfate in the collector tube C to absorb water formed in the pyrolysis.

7.2.1.4 Carry out a test for halogen, for instance by placing a strip of moistened indicator paper (5.5) across the mouth of the collecting tube. An acid colour, pH 1 to pH 2, indicates the presence of halogen. Residues of halogenated additives present in the vulcanizate may cause interference. Other suitable halogen-detection methods may also be used.

7.2.1.5 Bring the electric furnace D to 525 °C ± 50 °C and hold within this temperature range. This temperature range is recommended to obtain rapid pyrolysis without excessive degradation or carbonization. A temperature of 475 °C is advised, however, to obtain the maximum quantity of pyrolysate for NR, IR, BR, SBR, IIR, BIIR and CIIR.

7.2.1.6 After substituting the pyrolysis tube A containing prepared sample inside with nitrogen (5.1), pass a slow stream of nitrogen through the tube and into a hole in the aluminium block E. Nitrogen serves to displace air, prevent oxidation and facilitate transfer of the pyrolysis products into the collecting tube C. Maintain the nitrogen flow at 10 cm^3/min ± 2 cm^3/min.

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012

7.2.1.7 Continue the heating to complete distillation, i.e. for about 15 min.

7.2.1.8 Place a few drops of the homogenized pyrolysate between two potassium bromide salt plates and mount the cell in the infrared spectrometer. Run the spectrum immediately after pyrolysis to avoid oxidation.

7.2.1.9 Record the infrared spectrum from 4 000 cm^{-1} to 600 cm^{-1}, performing the same checks as described in 7.1.6.

7.2.2 Alternative method: Gas flame pyrolysis

7.2.2.1 Rapid pyrolysis may be performed in a test tube in place of the procedures described in 7.2.1.2, 7.2.1.3, 7.2.1.5, 7.2.1.6 and 7.2.1.7.

7.3 Procedure for vulcanized rubber film obtained after evaporation of the solution solvent

NOTE The methods described in 7.3.1 and 7.3.2 may give different relative absorbances for the polymers in a given blend. The films obtained by the method described in 7.3.2 may contain a higher proportion of the thermally less stable polymer.

7.3.1 Dissolution of vulcanizate

7.3.1.1 Prepare a test sample of about 2 g (or 6 g if the presence of chloroprene rubber is suspected) (see 7.3.1.2 and 7.3.1.3) and proceed with the extraction as described in ISO 1407.

7.3.1.2 Pyrolyse approximately 1 g of the prepared test sample and carry out a halogen test as described in 7.2.1.4.

7.3.1.3 If no chloroprene rubber is present, place 1 g of the test sample prepared in 7.3.1.1 and 50 cm^3 of a solvent appropriate to the rubber type (see 5.3) (1,2-dichlorobenzene is suggested) in a 100 cm^3 flask fitted with a reflux condenser. If chloroprene rubber is present, place approximately 5 g of the test sample prepared in 7.3.1.1 with 200 cm^3 of solvent in a 500 cm^3 flask fitted with a reflux condenser.

Heat the contents until the test sample has dissolved.

The time required for adequate dissolution varies depending on the rubber, e.g. 3 h to 4 h for NR; 12 h for CR. To reduce the risk of altering the molecular structure of the rubber, do not exceed 12 h heating.

7.3.1.4 If the rubber does not contain carbon black, centrifuge to eliminate mineral fillers.

7.3.1.5 If the rubber contains carbon black, add 10 g to 20 g of filter aid (6.7) and filter through ordinary filter paper. Should the filtrate contain carbon black, repeat the filtration with more filter aid.

NOTE Acrylonitrile-butadiene rubber (NBR) may be retained on the filter paper.

7.3.1.6 Concentrate the centrifuged or filtered solution to a small volume under a stream of nitrogen (5.1) or reduced pressure.

7.3.1.7 Evaporate a few drops of the concentrated solution on a potassium bromide salt plate.

7.3.1.8 Record the infrared spectrum from 4 000 cm^{-1} to 600 cm^{-1}, performing the same checks as described in 7.1.6.

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012

7.3.2 Mild thermal degradation of vulcanizates

7.3.2.1 This technique shall not be used on blends which may contain chloroprene rubber.

7.3.2.2 Prepare a test sample of 2 g as described in 7.2.1.1

7.3.2.3 Place the prepared test sample in a test tube capped with glass wool and heat for about 10 min in an oven (6.4) regulated at 200 °C ± 5 °C (a temperature of 180 °C is advised for NR, IR, BR, SBR, IIR, BIIR and CIIR rubbers).

7.3.2.4 Allow the test sample to cool, transfer to a 100 cm^3 flask fitted with a reflux condenser and add 50 cm^3 of chloroform (5.3.1) to the flask. Place the flask in a hot water bath.

7.3.2.5 Allow the flask and contents to remain for about 30 min in the water bath, with the solvent refluxing, to dissolve the degraded rubber.

7.3.2.6 Filter the mixture obtained in 7.3.2.5 through filter paper to remove any undissolved vulcanizate and fillers. Should carbon black be released from the vulcanizate, add a small amount of filter aid (6.7) to the solution before filtering.

7.3.2.7 When it is suspected that the filtrate obtained in 7.3.2.6 contains material other than rubber which might interfere in the interpretation of the final spectrum, precipitate the polymer from the filtrate obtained in 7.3.2.6 using methanol. Filter off the recovered polymer and redissolve it in chloroform (5.3.1).

7.3.2.8 Evaporate a few drops of the chloroform solution on a potassium bromide salt plate (6.6) to give a film thickness suitable for the production of an analytical spectrum.

7.3.2.9 Record the spectrum from 4 000 cm^{-1} to 600 cm^{-1}, performing the same checks as described in 7.1.6.

8 Procedure for attenuated total reflectance (ATR or micro-ATR) surface analysis

8.1 Carry out an extraction of the test sample in accordance with ISO 1407, using a suitable solvent, or clean the sample surface with the solvent, and then dry quickly to eliminate the solvent.

8.2 Clean the crystal surface before the test.

8.3 Place the test sample on the crystal, ensuring good contact.

8.4 Record the spectrum.

NOTE The presence of some mineral fillers may mask the polymer spectrum. Sometimes, they may be eliminated by specific chemical attacks from surface, under particular conditions.

9 Interpretation of spectra

9.1 Reference spectra

9.1.1 Due to the existence of different spectral presentation modes, it may be necessary to prepare a set of reference spectra on the same infrared spectrometer as is used to analyse the unknown samples.

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012

9.1.2 Reference spectra shall be produced from test samples of known composition, following the procedure used for unknown samples.

9.1.3 Spectra of mixtures are not given in Annex B because of the multiplicity of polymer combinations and proportions. Each laboratory should prepare its own set from test samples of known composition.

9.1.4 Small, but unavoidable, variations in experimental conditions and instrument characteristics may give rise to slight differences in spectra. Spectra produced at different times may not be identical in terms of peak height and absorbance.

9.1.5 In all cases, spectra shall be interpreted bearing in mind the result of the test for halogen.

9.1.6 The comparison between test spectra and reference spectra shall take into account the wavenumbers of the bands, how many bands are present, their relative intensity and their form. Any unexpected bands shall also be interpreted. It is essential that all the bands be examined, irrespective of their number.

9.2 Tables of diagnostic absorptions

9.2.1 The tables of diagnostic absorption bands given in Annex B shall be used only in conjunction with reference spectra. Their purpose is to indicate the principal absorption bands.

9.2.2 The tables complement the reference spectra by drawing attention to absorption bands which are absent, permitting the elimination of certain rubbers when ambiguity could otherwise arise.

9.2.3 Diagnostic absorption bands are classified by increasing wavenumber. A diagnostic absorption band is one whose features are recognized by an experienced analyst as being of significance in rubber identification. These features, associated with certain compositional or structural characteristics of the pyrolysates and films, are reproducible in the sense that they are not seriously influenced by moderate variations in the conditions of pyrolysis or of dissolution.

10 Test report

The test report shall include the following particulars:

a) sample details:

   1) a full description of the sample,

   2) if appropriate, the method of preparation of the test sample from the sample;

b) test method:

   1) a reference to this International Standard, i.e. ISO 4650,

   2) the test procedure used (film or ATR);

c) details of any procedures not specified in this International Standard;

d) the test results, i.e. identification of the rubber(s) in the sample;

e) the date of the test.

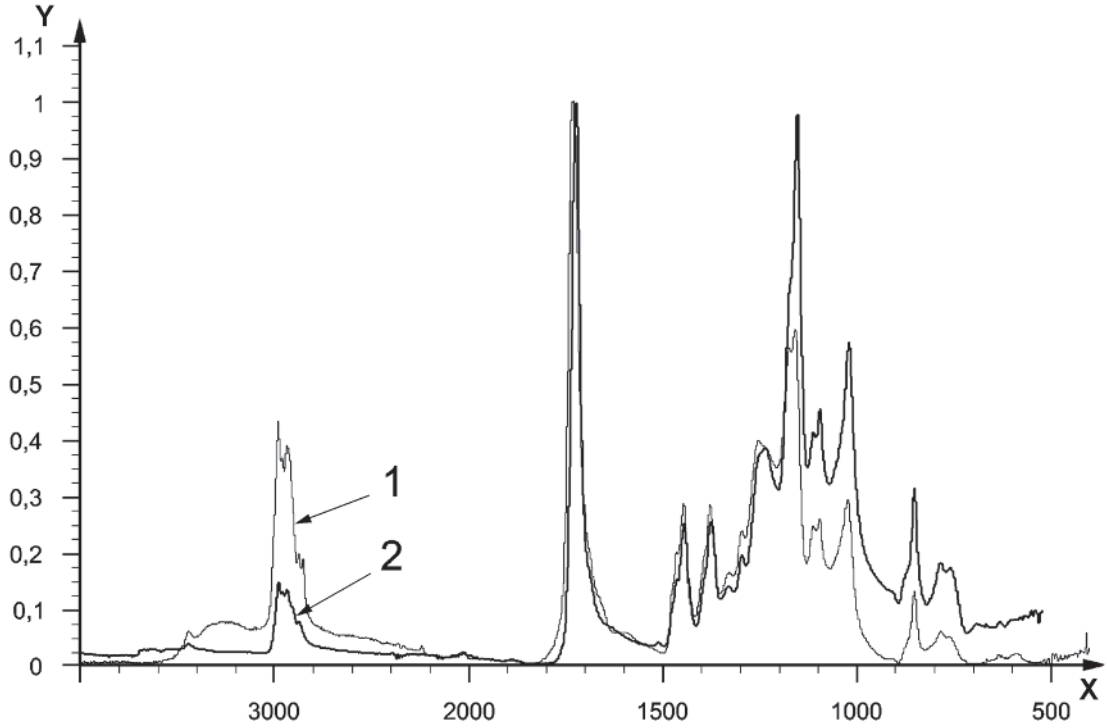
© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012

Annex A
(informative)

Comparison of reflectance and transmission spectra

Spectra resulting from reflectance (ATR) and transmission (film) differ only in the intensity of the absorption bands. Figure A.1 shows a comparison of the two types of spectrum.



Key

X wave number, cm^{-1}

Y absorbance, %

1 transmission (film) spectrum

2 ATR spectrum

Figure A.1 — Acrylic rubber (ACM) — Comparison of transmission (film) and reflectance (ATR) spectra

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012

Annex B
(informative)

Absorption characteristics and reference spectra

B.1 General

B.1.1 This annex provides tables of absorption characteristics and figures showing reference spectra for pyrolysates and films.

B.1.2 Comparisons between sample spectra and reference spectra will have to take into account the position of the bands, how many there are, their relative intensity and their shape.

B.1.3 It is essential that all the bands in a spectrum be examined, with no restrictions on the zone which is searched for characteristic bands.

B.2 Tables of absorption characteristics and figures showing reference spectra

B.2.1 In order to ease the task of the user of this International Standard, the scale chosen for the presentation of the spectra is designed to show clearly the specific absorption bands.

B.2.2 Table B.1 indicates which figures correspond to the reference spectra for which types of rubber.

NOTE Absorption by carbon dioxide (in the neighbourhood of 2 350 cm^{-1} and 670 cm^{-1}) and by water molecules (4 000 cm^{-1} to 3 500 cm^{-1} and 2 000 cm^{-1} to 1 500 cm^{-1}) can also be observed in the spectra.

Table B.1 — Types of rubber and corresponding reference spectra

Table number	Symbol for rubber	Type of rubber	Figure number		
			Raw polymer		Vulcanizate pyrolysate
			Film	ATR	
M group					
B.2	ACM	Acrylic rubber	B.1 a)	B.1 b)	B.2
B.3	AEM	Acrylic and ethylene rubber	B.3 a)	B.3 b)	B.4
B.4	CM	Chloropolyethylene	B.5 a)	B.5 b)	B.6
B.5	CSM	Chlorosulfonylpolyethylene	B.7 a)	B.7 b)	B.8
B.6	EPDM	Ethylene-propylene-diene terpolymer	B.9 a)	B.9 b)	B.10
B.7	FKM	Fluorocarbon rubber	B.11 a)	B.11 b)	B.12
O group					
B.8	CO	Polychloromethyloxirane	B.13 a)	B.13 b)	B.14
B.9	ECO	Copolymer of ethylene oxide and chloromethyl-oxirane	B.15 a)	B.15 b)	B.16
B.10	GECO	Terpolymer of epichlorohydrin-ethylene oxide-allyl glycidyl ether	B.17 a)	B.17 b)	B.18
Q group					
B.11	MQ	Polydimethylsiloxane	B.19 a)	B.19 b)	B.20
B.12	PMQ	Polyphenylmethylsiloxane	B.21 a)	B.21 b)	B.22
B.13	FMQ	Polymethylfluorosiloxane	B.23 a)	B.23 b)	B.24
R group					

AVA Documents
Document Sharing Hub

© ISO 2012 – All rights reserved

Full document details: BS ISO 4650:2012

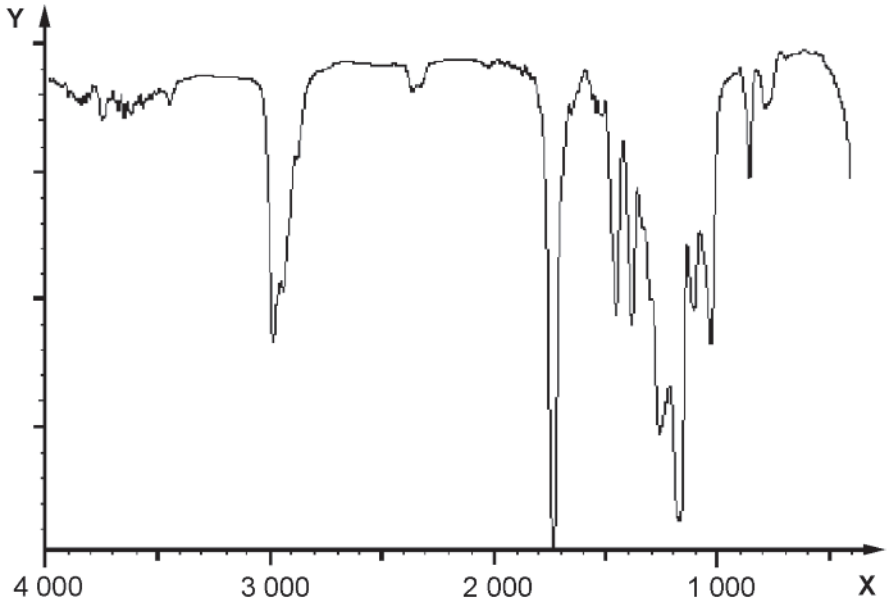
Table B.1 *(continued)*

Table number	Symbol for rubber	Type of rubber	Figure number		
			Raw polymer		Vulcanizate pyrolysate
			Film	ATR	
B.14	BR	Butadiene rubber			
		high-*cis* BR	B.25 a)	B.25 b)	B.26
		high-*trans* BR	B.27	—	
		low-*cis* BR	B.28	—	
B.15	CR	Chloroprene rubber	B.29 a)	B.29 b)	B.30
B.16	IIR	Isobutene-isoprene rubber	B.31 a)	B.31 b)	B.32
B.17	BIIR	Bromo-isobutene-isoprene rubber	B.33	—	B.34
B.18	BIMS	Brominated isobutylene-co-*p*-methylstyrene	B.35 a)	B.35 b)	B.36
B.19	NR	Natural rubber	B.37 a)	B.37 b)	B.38
B.20	IR	Synthetic isoprene rubber			
		high-*cis* IR	B.39 a)	B.39 b)	B.40
		high-*trans* IR	B.41	—	B.42
B.21		3,4 IR	B.43 a)	B.43 b)	B.44
B.22	NBR	Acrylonitrile-butadiene rubber	B.45 a)	B.45 b)	B.46
B.23	HNBR	Hydrogenated acrylonitrile-butadiene rubber	B.47 a)	B.47 b)	B.48
B.24	XNBR	Carboxylic-acrylonitrile-butadiene rubber	B.49 a)	B.49 b)	B.50
B.25	NBR/PVC	Acrylonitrile-butadiene rubber associated with PVC	B.51 a)	B.51 b)	B.52
B.26	SBR	Styrene-butadiene rubber		B.53	
	E-SBR	Emulsion-polymerized SBR			
		23,5 % styrene E-SBR	B.54	—	B.55
		high-styrene E-SBR	B.56	—	
	S-SBR	Solution-polymerized SBR			
		high-vinyl S-SBR	B.57	—	
		high-styrene S-SBR	B.58	—	
B.27	HSBR	Hydrogenated styrene-butadiene rubber	B.59	—	
T group					
B.28	EOT	Polysulfide		B.60	B.61
U group					
B.29	AU	Polyesterurethane	B.62 a)	B.62 b)	B.63
B.30	EU	Polyetherurethane	B.64 a)	B.64 b)	B.65
TFE group					
B.31	TPS-SBS	Block copolymer of styrene and butadiene	B.66		
B.32	TPS-SEBS	Polystyrene-poly(ethylene-butylene)-polystyrene	B.67		
B.33	TPS-SIS	Block copolymer of styrene and isoprene	B.68		
B.34	TPS-SEPS	Polystyrene-poly(ethylene-propylene)-polystyrene	B.69		
B.35	TPZ	Syndiotactic poly(1,2-butadiene)	B.70		
B.36	TPC-EE	Copolyester TPE with a soft segment with ester and ether linkages	B.71		

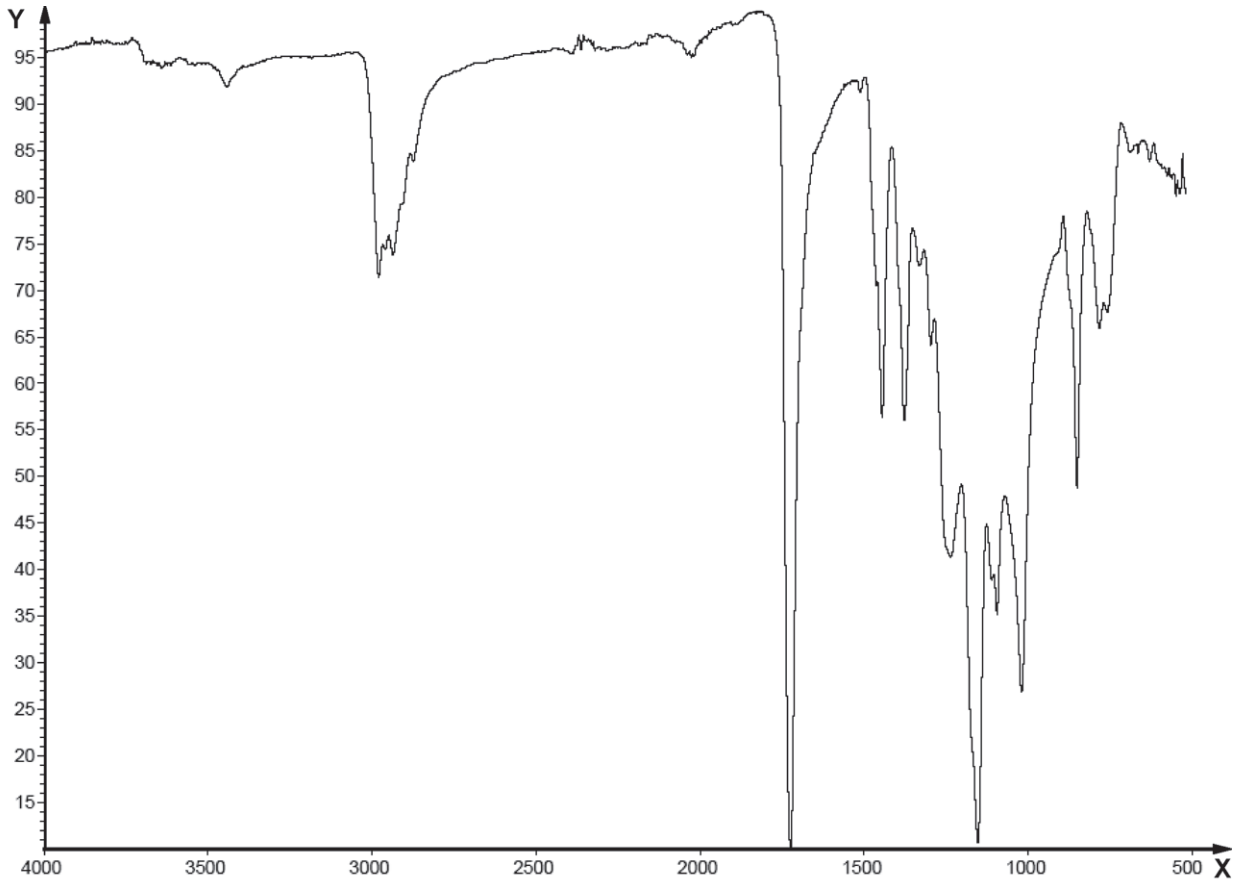
Table B.2 — Acrylic rubber (ACM)

Raw polymer		Pyrolysate (vulcanizate)	
Wave number cm^{-1}	**Functional group**	**Wave number** cm^{-1}	**Functional group**
1 150 to 1 260	$-\overset{\vert}{\underset{\vert}{C}}-O-$	1 150 to 1 260	$-\overset{\vert}{\underset{\vert}{C}}-O-$
1 740	> C = O	1 740	> C = O

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



a) Film



b) ATR

Key

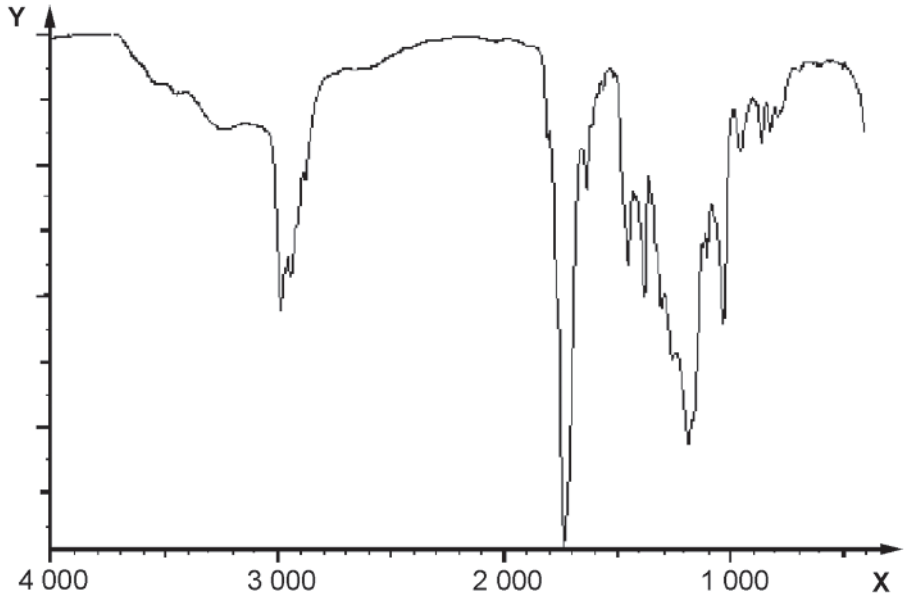
X wave number, cm^{-1}

Y transmittance, %

Figure B.1 — Acrylic rubber — Raw polymer

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



Key

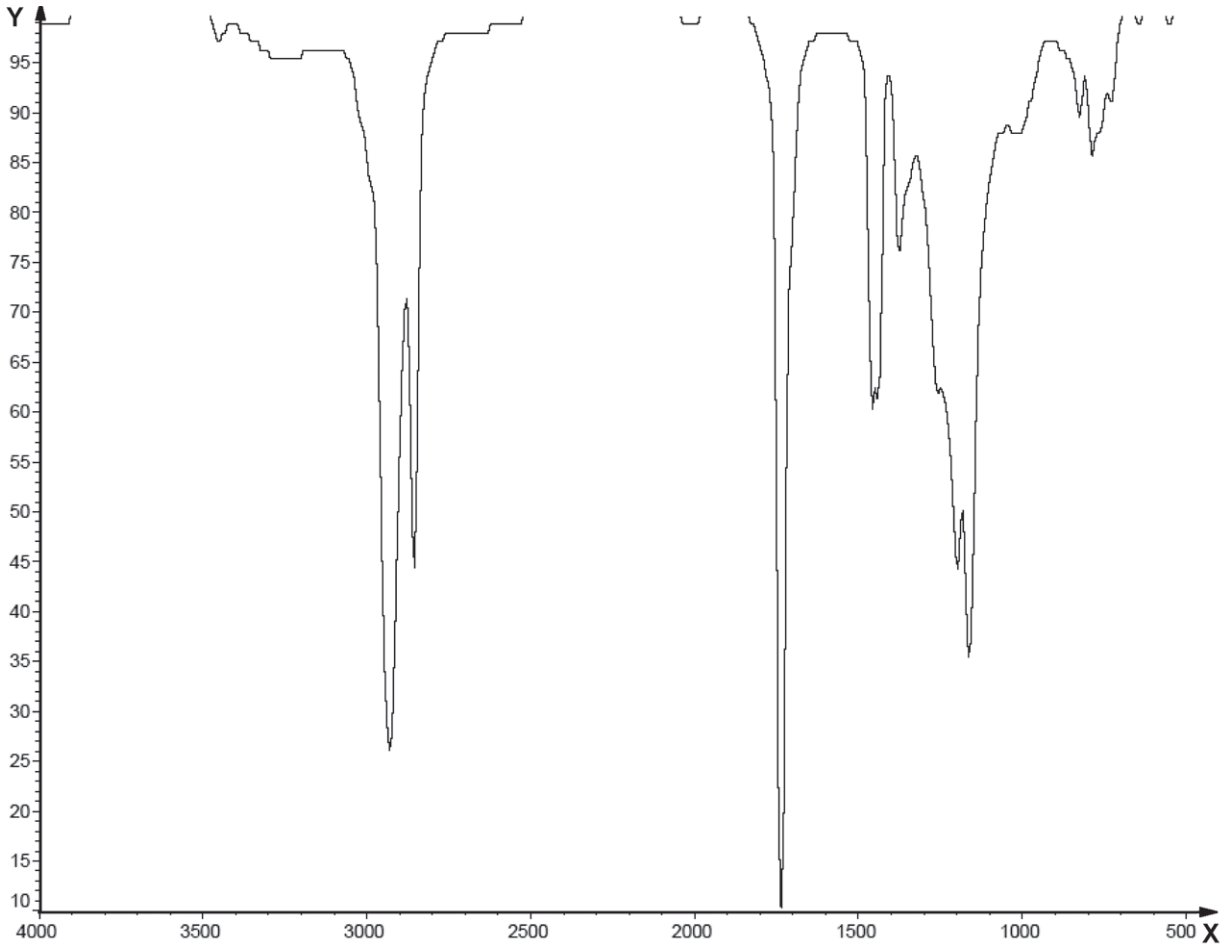
X wave number, cm^{-1}
Y transmittance, %

Figure B.2 — Acrylic rubber — Vulcanizate

Table B.3 — Acrylic and ethylene rubber (AEM)

Raw polymer		Pyrolysate (vulcanizate)	
Wave number cm^{-1}	**Functional group**	**Wave number** cm^{-1}	**Functional group**
725	$— (CH_2)_n —$		
1 150 to 1 260	$-\overset{\vert}{\underset{\vert}{C}}-O-$	1 150 to 1 260	$-\overset{\vert}{\underset{\vert}{C}}-O-$
1 460	$— CH_2 —$	1 460	$— CH_2 —$
1 740	$> C = O$	1 740	$> C = O$

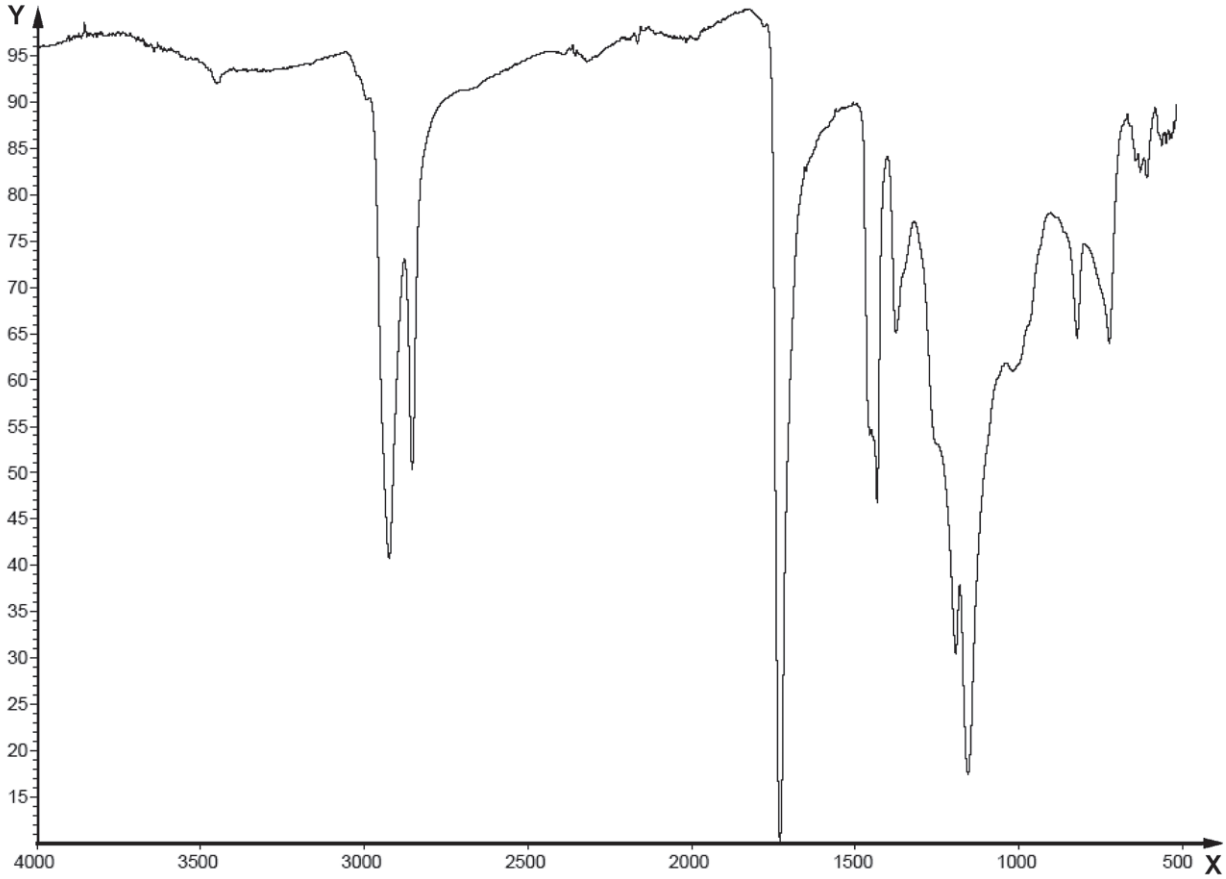
© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



a) Film

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



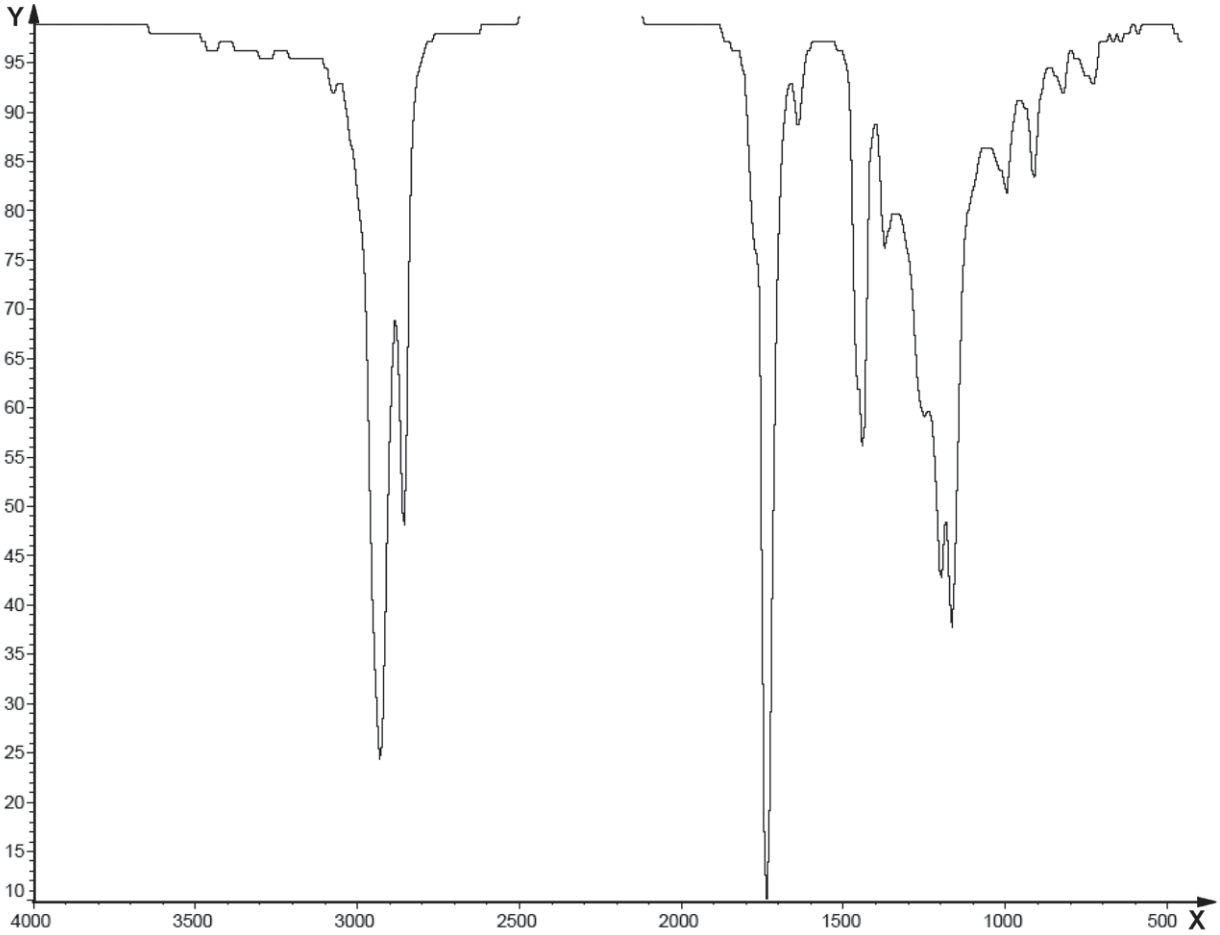
b) ATR

Key

X wave number, cm^{-1}
Y transmittance, %

Figure B.3 — Acrylic and ethylene rubber — Raw polymer

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



Key

X wave number, cm^{-1}

Y transmittance, %

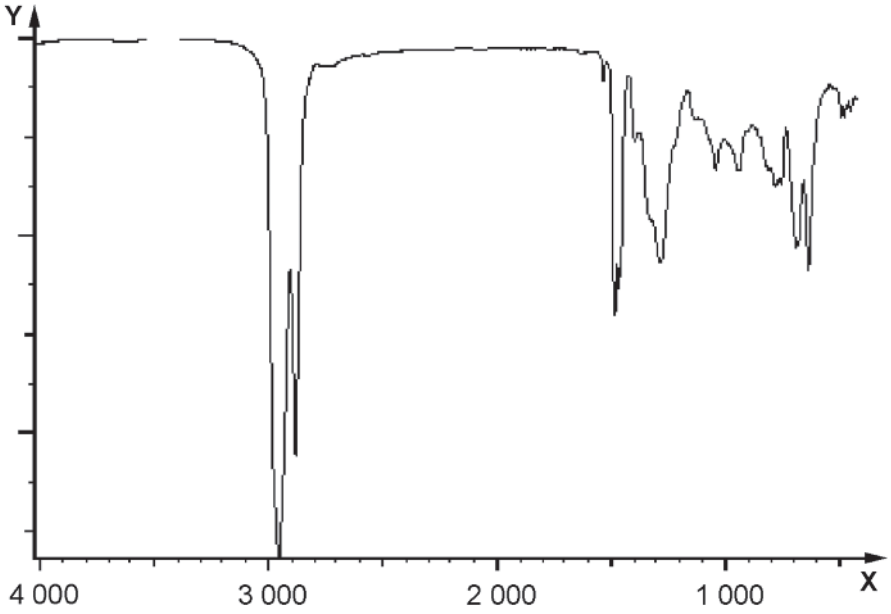
Figure B.4 — Acrylic and ethylene rubber — Vulcanizate

Table B.4 — Chloropolyethylene (CM)

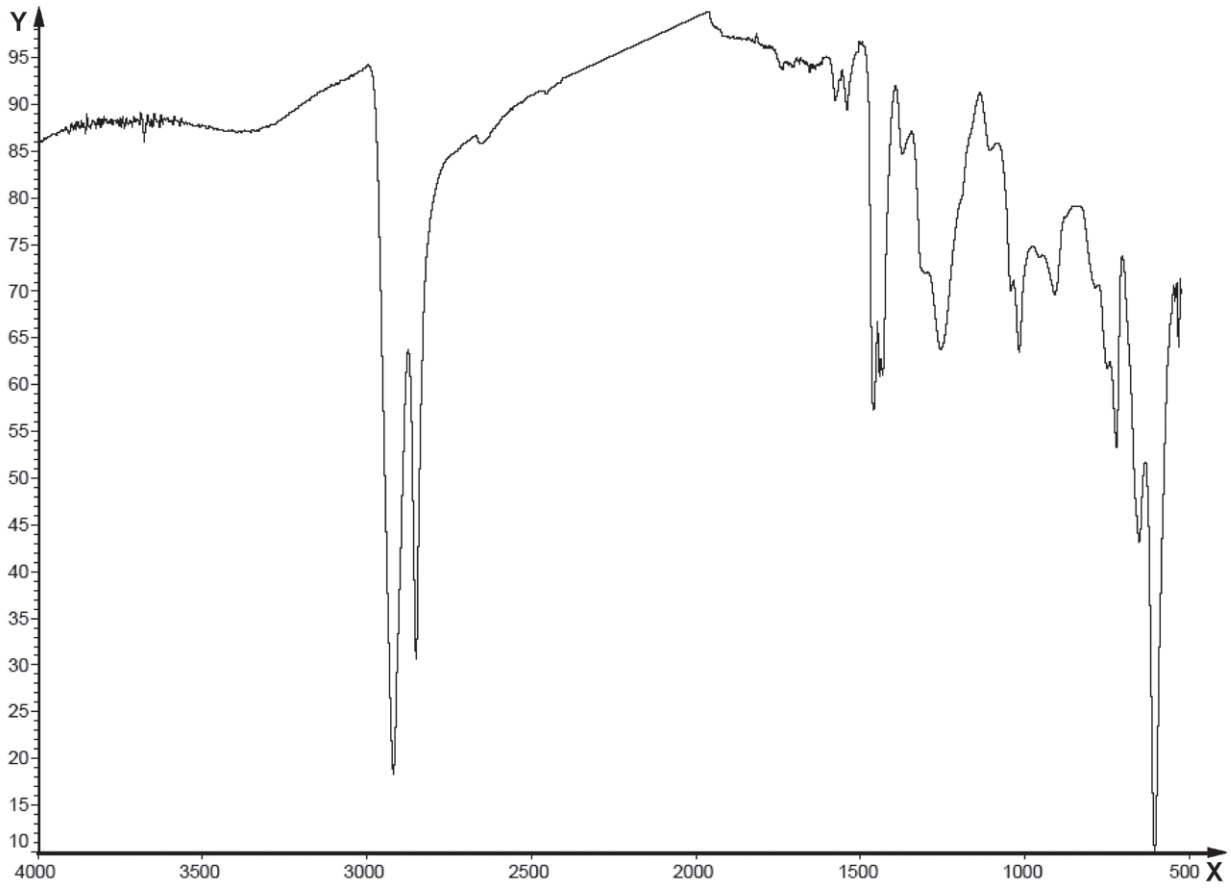
Raw polymer		**Pyrolysate (vulcanizate)**	
Wave number cm^{-1}	**Functional group**	**Wave number** cm^{-1}	**Functional group**
600 to 700	$-\overset{\vert}{\underset{\vert}{C}}-Cl-$	600 to 700	$-\overset{\vert}{\underset{\vert}{C}}-Cl-$
720	$(CH_2)_n$	820 to 890	$> C = CH_2$
		910	$— CH = CH_2$
		970	$— CH = CH —$ (*trans*)
		990	$— CH = CH_2$
		1 370	CH_3
1 460	$— CH_2 —$	1 460	$— CH_2 —$
NOTE The spectrum varies very considerably with changes in chlorine content.			

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



a) Film



b) ATR

Key

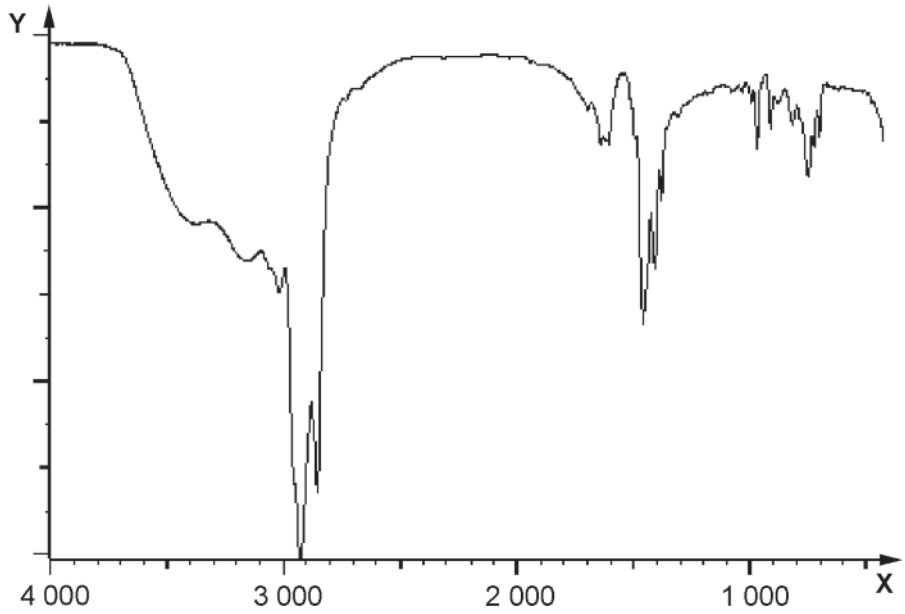
X wave number, cm^{-1}

Y transmittance, %

Figure B.5 — Chloropolyethylene — Raw polymer

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



Key

X wave number, cm^{-1}

Y transmittance, %

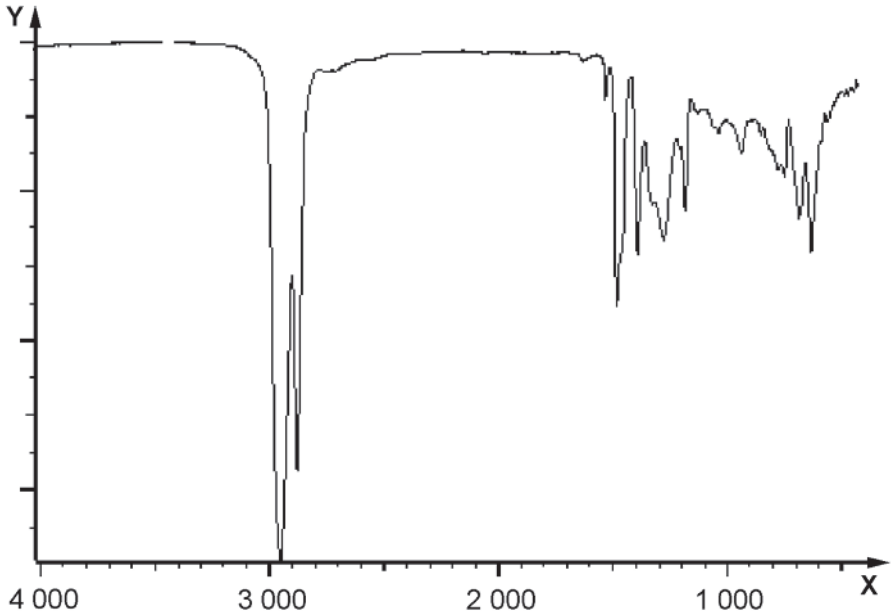
Figure B.6 — Chloropolyethylene — Vulcanizate

Table B.5 — Chlorosulfonylpolyethylene (CSM)

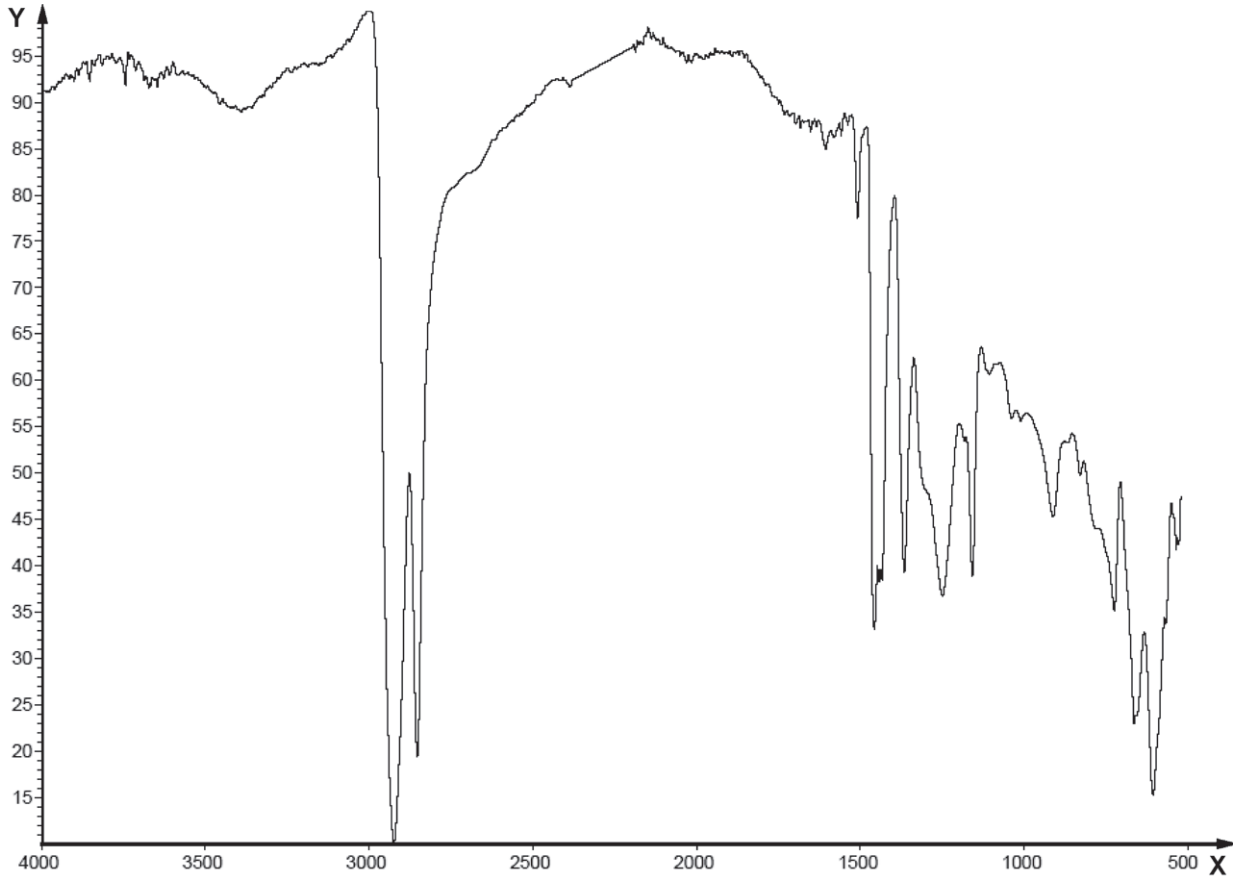
Raw polymer		Pyrolysate (vulcanizate)	
Wave number cm^{-1}	**Functional group**	**Wave number** cm^{-1}	**Functional group**
720	$(CH_2)_n$	695	
		720	$(CH_2)_n$
		740	$-\overset{\vert}{\underset{\vert}{C}}-Cl-$
		815	
		910	$— CH = CH_2$
1 160	$— SO_2Cl$	970	$— CH = CH —$ (*trans*)
1 260		990	$— CH = CH_2$
1 370	$— CH_3$		
1 460	$— CH_2 —$	1 460	$— CH_2 —$

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



a) Film



b) ATR

Key

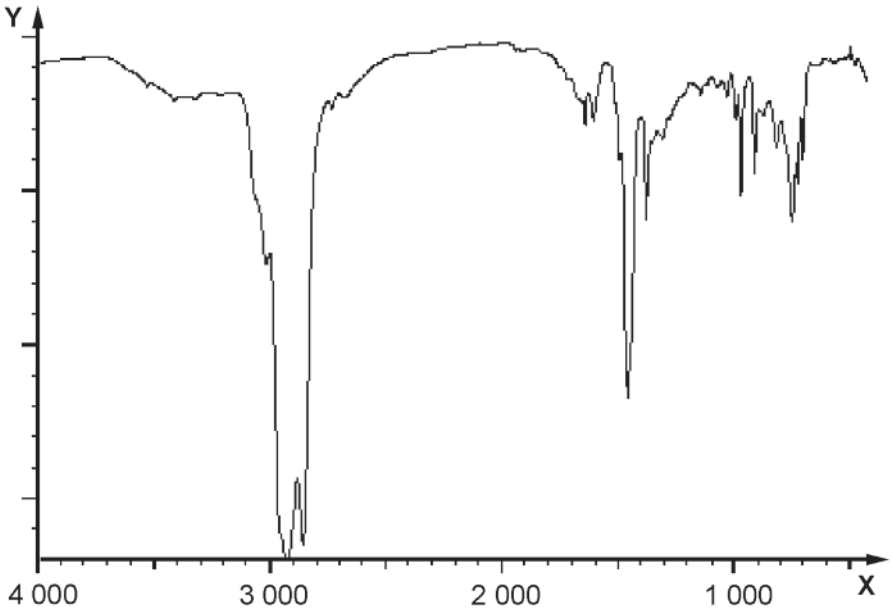
X wave number, cm^{-1}

Y transmittance, %

Figure B.7 — Chlorosulfonylpolyethylene — Raw polymer

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



Key

X wave number, cm^{-1}

Y transmittance, %

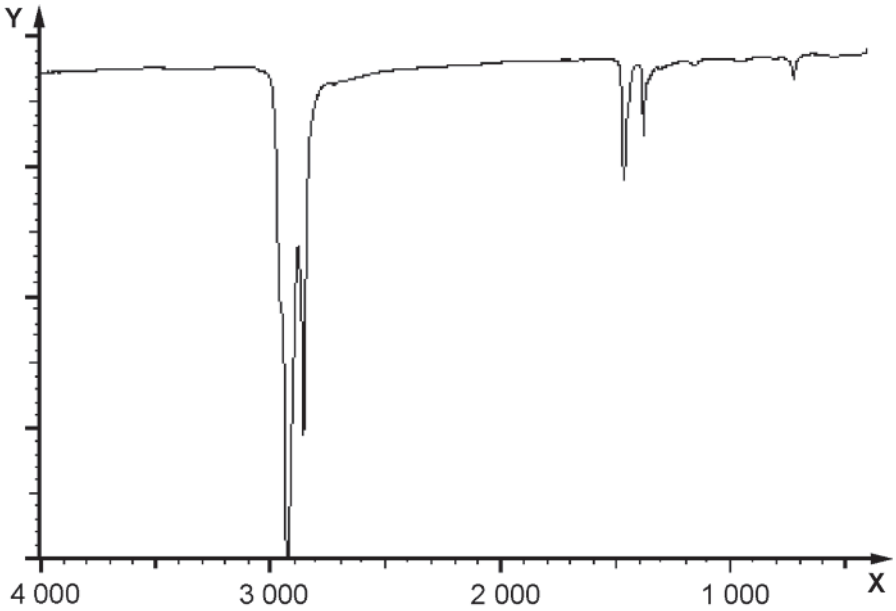
Figure B.8 — Chlorosulfonylpolyethylene — Vulcanizate

Table B.6 — Ethylene-propylene-diene terpolymer (EPDM)

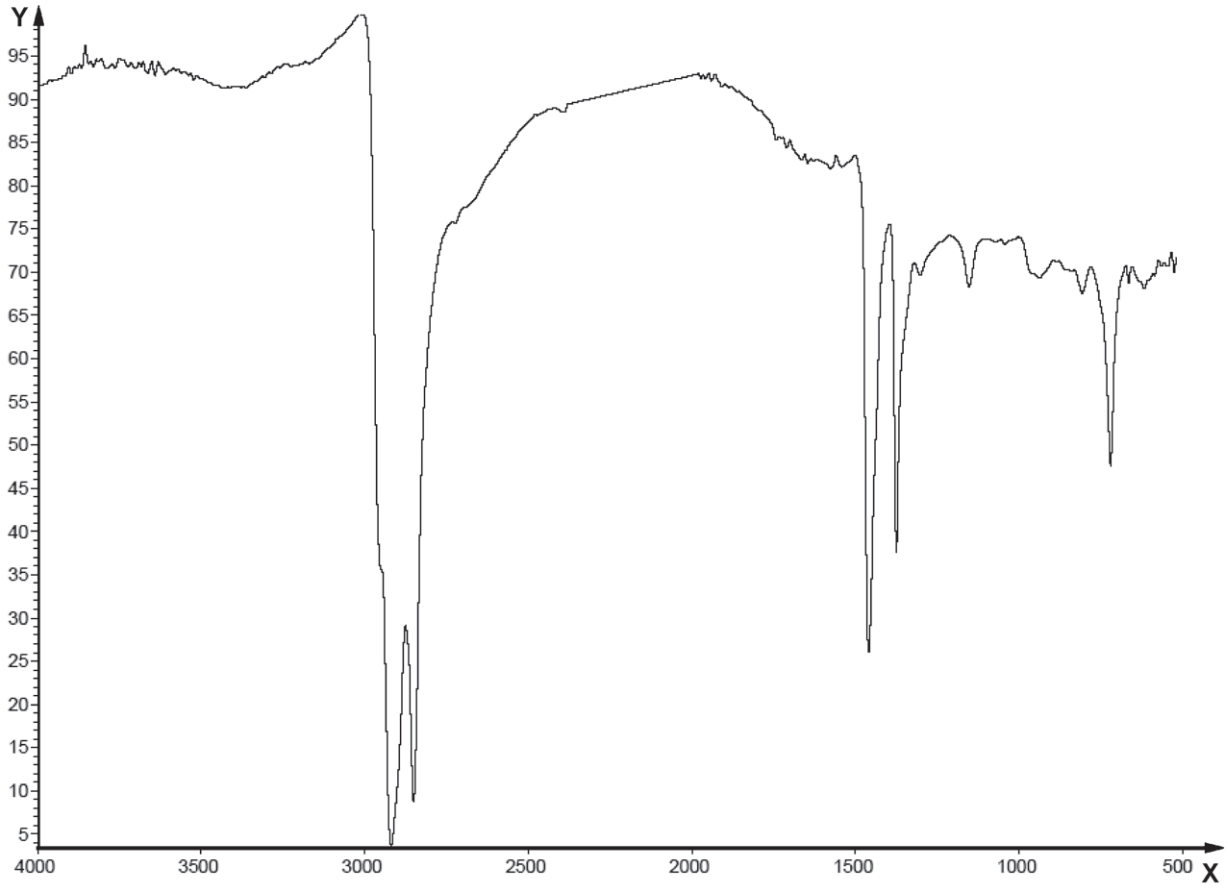
Raw polymer		Pyrolysate (vulcanizate)	
Wave number cm^{-1}	**Functional group**	**Wave number** cm^{-1}	**Functional group**
720	$(CH_2)_n$	720	$(CH_2)_n$
		890	$> C = CH_2$
		910	$— CH = CH_2$
		970	$— CH = CH —$ (*trans*)
		990	$— CH = CH_2$
1 370	$— CH_3$	1 370	$— CH_3$
1 460	$— CH_2 —$	1 460	$— CH_2 —$

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



a) Film



b) ATR

Key

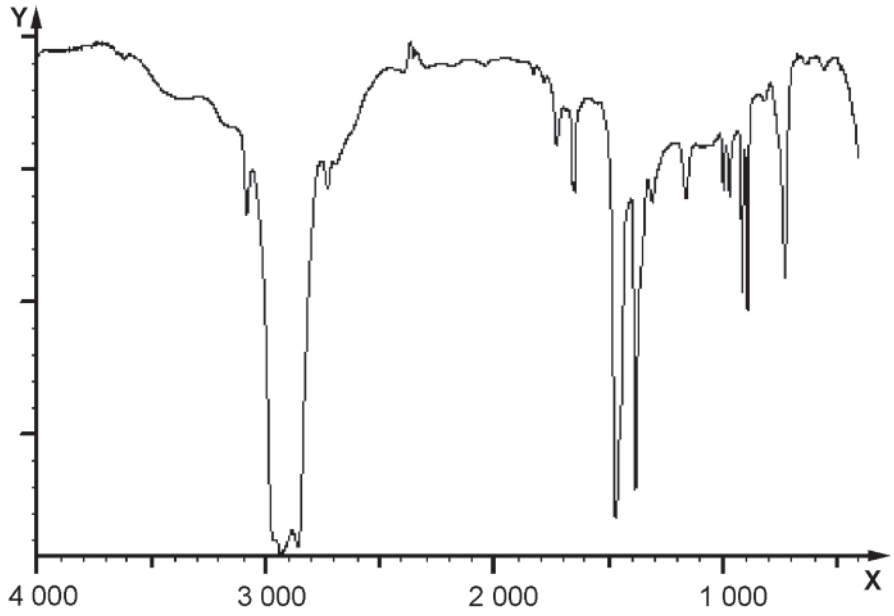
X wave number, cm^{-1}

Y transmittance, %

Figure B.9 — Ethylene-propylene-diene terpolymer — Raw polymer

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



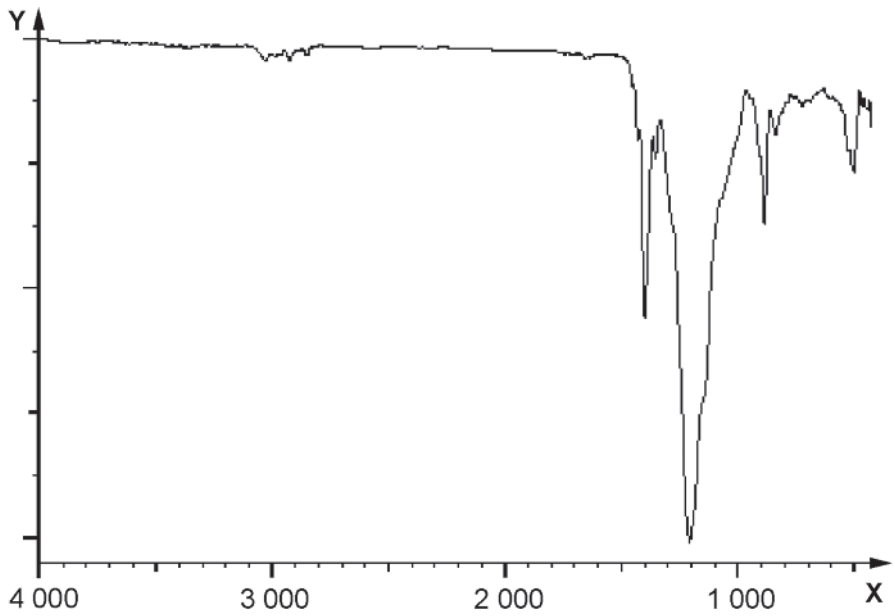
Key

X wave number, cm^{-1}
Y transmittance, %

Figure B.10 — Ethylene-propylene-diene terpolymer — Vulcanizate

Table B.7 — Fluorocarbon rubber (FKM)

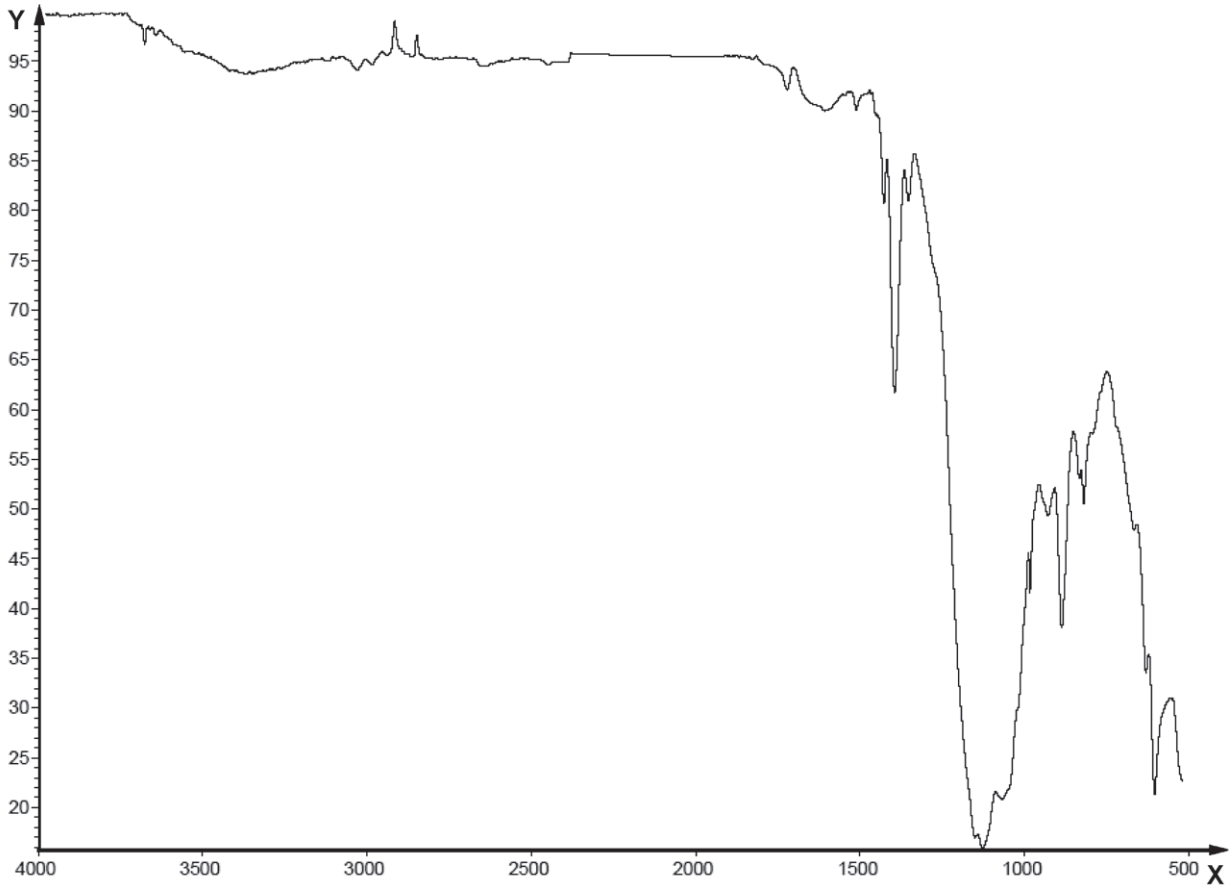
Raw polymer		Pyrolysate (vulcanizate)	
Wave number cm^{-1}	**Functional group**	**Wave number** cm^{-1}	**Functional group**
1 000 to 1 400	$-\overset{\vert}{\underset{\vert}{C}}-F$	1 000 to 1 400	$-\overset{\vert}{\underset{\vert}{C}}-F$



a) Film

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012



b) ATR

Key

X wave number, cm^{-1}

Y transmittance, %

Figure B.11 — Fluorocarbon rubber — Raw polymer

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 4650:2012