
International Standard



4650

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Rubber — Identification — Infra-red spectrometric method

Caoutchouc — Identification — Méthode spectrométrique dans l'infrarouge

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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 developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4650 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in September 1982.

It has been approved by the member bodies of the following countries:

Australia	Hungary	South Africa, Rep. of
Austria	India	Spain
Belgium	Italy	Sri Lanka
Canada	Korea, Rep. of	Sweden
China	Malaysia	Thailand
Czechoslovakia	Netherlands	Turkey
Egypt, Arab Rep. of	New Zealand	United Kingdom
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No member body expressed disapproval of the document.

Rubber — Identification — Infra-red spectrometric method

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1 Scope

1.1 This International Standard specifies a method for the identification of rubber polymers, based on infra-red spectrometric examination of both pyrolysis products (pyrolysates) and of films cast from solutions. Typical spectra can be found in the annex.

The use of attenuated total reflectance infra-red spectroscopy is not included.

1.2 The principle of the method implies that sample preparation, and preparation and analysis of the infra-red spectra, have to be carried out by experienced personnel and that the equipment used for the production of the spectra has to be operated according to the manufacturer's instructions for optimum performance. No details for the operation of infra-red spectrometers are included, however, in this International Standard.

1.3 The method specified is not intended for quantitative analysis.

2 Field of application

2.1 General

The method is applicable to rubbers in the raw state and, if compounded, in both the cured and uncured states. The method is applicable to the following types of rubbers, occurring either alone or in a binary mixture when the proportion of the minor component is, in general, not less than 20 % (*m/m*) of the mixture (see the exceptions in 2.2).

NOTE — An extension of this method to other polymers not covered at present is under consideration.

2.1.1 Isoprene rubbers

2.1.1.1 Natural rubber, synthetic polyisoprene, gutta percha and balata are included under 2.1.1.

2.1.1.2 Examination of the pyrolysate will not distinguish between the different forms of polyisoprene.

2.1.1.3 Examination of a film will distinguish natural and synthetic *cis*-polyisoprenes from balata, gutta percha and synthetic *trans*-polyisoprenes.

2.1.1.4 The method will not distinguish between natural rubber and synthetic *cis*-polyisoprenes.

2.1.1.5 The method will not distinguish between balata, gutta percha and synthetic *trans*-polyisoprenes.

2.1.2 Styrene-butadiene rubbers

2.1.2.1 For the purpose of this International Standard, all random copolymers of styrene and butadiene and their oil-extended forms are included under 2.1.2.

2.1.2.2 Examination of the pyrolysate will not distinguish between emulsion- and solution-polymerized rubbers.

2.1.2.3 Examination of the pyrolysate or film may give some information about the monomer ratio and the polymerization system.

2.1.3 Butadiene-acrylonitrile rubbers

2.1.3.1 Examination of the pyrolysate will not measure the ratio of acrylonitrile to butadiene.

2.1.3.2 Examination of the pyrolysate or film may give some information about the acrylonitrile content.

2.1.4 Chloroprene rubbers

This method will not distinguish between the different types of chloroprene rubber.

2.1.5 Isobutene-isoprene rubbers (butyl)

This method will not distinguish between butyl rubber and its halogenated forms.

2.1.6 High *cis*-polybutadiene rubbers

2.1.6.1 Examination of the pyrolysate will not distinguish between polybutadiene rubbers having different isomer ratios.

2.1.6.2 Examination of a film may give some information about the isomer ratio and the monomer ratio.

2.1.7 Ethylene-propylene rubbers

This method will not distinguish between rubbers having different ratios of ethylene to propylene or between copolymers and terpolymers.

2.1.8 Chlorosulfonated polyethylene rubbers

This method will not distinguish between the different types of chlorosulfonated polyethylene rubbers.

2.2 Exceptions for blends

2.2.1 Analysis of a blend of natural and/or synthetic polyisoprene (20 %) and chloroprene (80 %) may present difficulties, and identification of the minor component may only be achieved when its content is equal to or greater than 30 % in the blend.

2.2.2 Analysis of a blend of styrene-butadiene rubber (80 %) and high *cis*-polybutadiene rubber (20 %) may present difficulties, and identification of the minor component may only be achieved when its content is equal to or greater than 30 % in the blend. A blend of styrene-butadiene rubber and polybutadiene rubber may give a spectrum indistinguishable from that of a styrene-butadiene rubber with a lower styrene content.

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

2.3 Special cases

In difficult cases, infra-red spectrometric examination of the gaseous pyrolysis products may give further information.

2.4 Reference spectra

Tables of diagnostic absorptions and reference spectra for the rubbers listed in 2.1 are given in the annex.

3 Reference

ISO 1407, *Rubber — Determination of solvent extract.*

4 Principle

4.1 Identification from pyrolysates

4.1.1 A small quantity of extracted and dried rubber is either pyrolysed in a stream of nitrogen in a test-tube held in a small, electric, thermoregulated furnace at 550 ± 25 °C (preferred method) or, alternatively, rapidly pyrolysed in a small test-tube held in the hot zone of a gas flame.

4.1.2 A test for halogen is conducted during pyrolysis.

4.1.3 A few drops of the pyrolysate are transferred to a salt plate and an infra-red spectrum is recorded over the wavelength range 2,5 to 15 μm (wavenumber range 4 000 to 667 cm^{-1}).

4.1.4 The rubber, whether raw or vulcanized, is identified by comparison with reference spectra and by reference to a table of diagnostic absorptions (significant absorptions expressed as either wavelengths or wavenumbers).

4.1.5 Each laboratory employing this International Standard should prepare spectra of all the rubbers they may be expected to identify. These spectra shall be prepared in the same manner, and using the same equipment, as will be used for unknown samples.

4.2 Identification of vulcanizates from films cast from solutions

Two alternative methods are described.

4.2.1 A small quantity of extracted and dried rubber is dissolved in 1,2-dichlorobenzene and filtered. A film is cast on a salt plate and its infra-red spectrum is recorded over the wavelength range 2,5 to 15 μm . The rubber is identified as in 4.1.4.

4.2.2 A small quantity of extracted and dried rubber is subjected to mild thermal degradation at 200 ± 5 °C for a short time. The degraded sample is dissolved in 1,1,1-trichloroethane and then dichloromethane. A film is cast on a salt plate and its infra-red spectrum is recorded over the wavelength range 2,5 to 15 μm . The rubber is identified as in 4.1.4.

4.3 Identification of raw polymers from films cast from solutions

A small quantity of extracted and dried rubber is dissolved in dichloromethane and filtered. A film is cast on a salt plate and its infra-red spectrum is recorded over the wavelength range 2,5 to 15 μm . The rubber is identified as in 4.1.4.

5 Reagents

All recognized health and safety precautions shall be in effect when carrying out this procedure. All evaporations shall be carried out in a fume cupboard (hood).

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Nitrogen, in pressurized cylinders.

5.2 Solvents for extraction (see 7.1.1).

NOTE — The solvents should be selected to achieve maximum extraction. Alternative solvents may be used if the analyst is sure that these do not interfere with the interpretation of the infra-red spectrum.

5.2.1 Methanol (preferred).

5.2.2 2-Propanol.

5.2.3 Ethanol or denatured ethanol.

5.2.4 Butanone.

5.2.5 Acetone.

5.2.6 Ethanol-toluene azeotrope.¹⁾

5.3 Solvents for rubber dissolution and film preparation.

NOTE — Alternative solvents may be used if the analyst is sure that these do not interfere with the interpretation of the infra-red spectrum.

5.3.1 Toluene.

5.3.2 1,1,1-trichloroethane.

5.3.3 Dichloromethane.

5.3.4 1,2-dichlorobenzene.

5.4 Sodium sulfate, anhydrous.

5.5 Congo red indicator paper.

6 Apparatus

Ordinary laboratory apparatus and

6.1 Extraction apparatus.

The apparatus specified in ISO 1407 is satisfactory.

6.2 Pyrolysis apparatus (see figure 1).

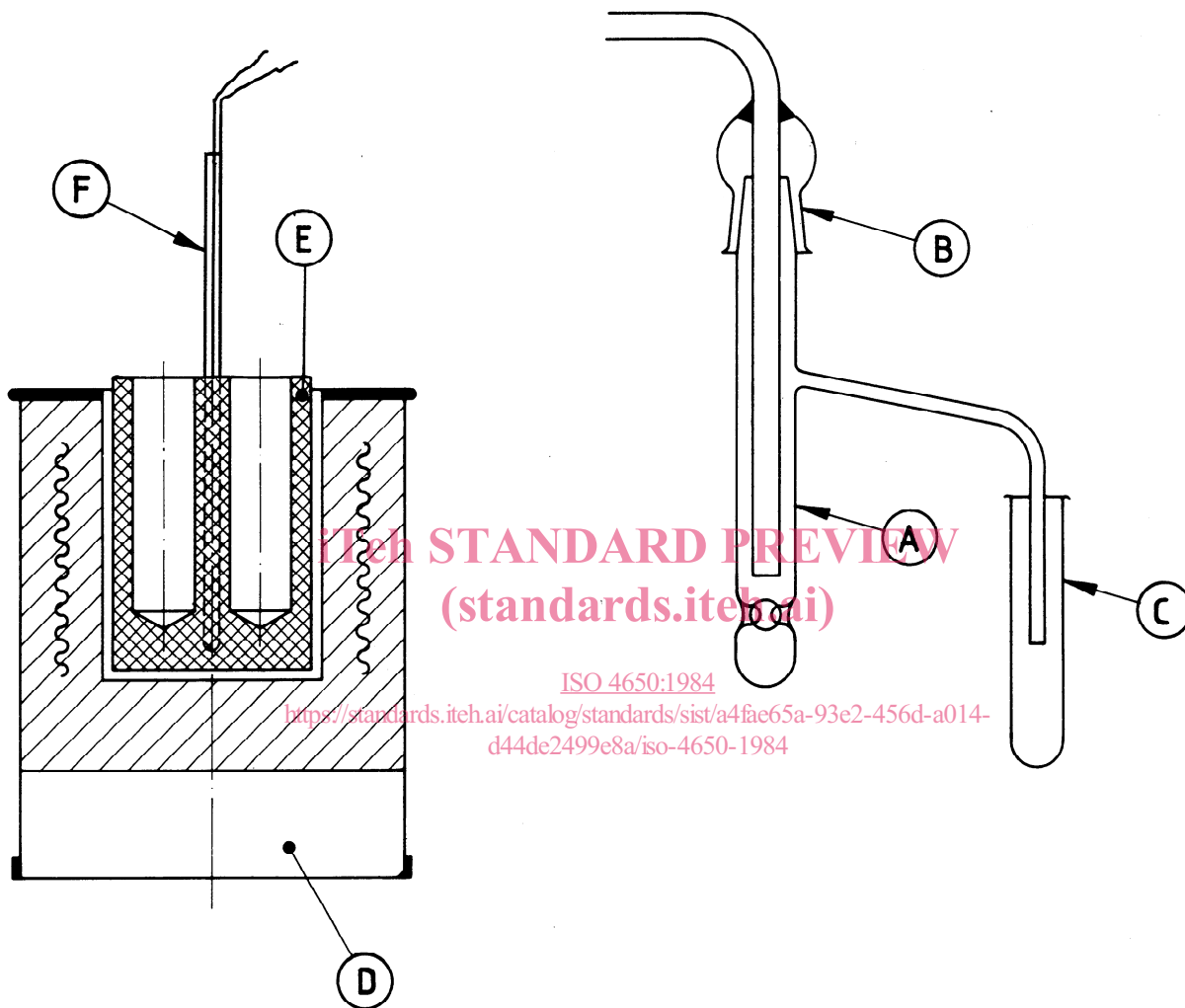
The pyrolysis apparatus consists of a glass tube (A) having inward projections to prevent the sample from falling to the bottom of the tube and a lateral condenser tube. The tube (A) has a ground glass standard joint (B) which carries a small glass adductor tube. A collecting tube (C) is placed under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

6.3 Capillary pipettes.

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

6.5 Boiling water-bath.

1) Mixture of 70 parts by volume of ethanol and 30 parts by volume of toluene. Reflux for 4 h over freshly calcined calcium oxide. Distil and collect a middle fraction with a boiling range of not more than 1 °C. If absolute ethanol is used, the drying over calcium oxide may be omitted.



- | | | | |
|-----|---------------------------------|-----|--------------------------------------|
| (A) | Glass tube for sample container | (D) | Electric, thermoregulated furnace |
| (B) | Ground glass joint | (E) | Aluminium block, bored to hold tubes |
| (C) | Collecting tube | (F) | Thermocouple |

Figure 1 – Temperature-controlled pyrolysis apparatus

6.6 Polished salt plates, of either sodium chloride (NaCl) or potassium bromide (KBr).

6.7 Filter aid, for example, diatomaceous silica, or any similar substitute.

6.8 Double-beam infra-red spectrometer, capable of recording a spectrum over the wavelength range 2,5 to 15 μm (see 1.2).

7 Procedure for pyrolysates

NOTE — The methods described in 7.1 and 7.2 may give different relative peak intensities for the polymers in a given blend.

7.1 Temperature-controlled pyrolysis in a stream of nitrogen (see figure 1) (Preferred method)

7.1.1 Mill the rubber into a thin sheet on a laboratory mill by the method described in ISO 1407, or, if this is not practicable, cut the rubber into cubes of maximum side 2 mm. Wrap approximately 0,5 g in filter paper or nylon cloth. Extract the wrapped test portion of raw rubber or vulcanizate in an extraction cup of the extraction apparatus (6.1) for a minimum of 16 h with the methanol or other appropriate solvent (see 5.2.1). The ethanol-toluene azeotrope (5.2.6) may be used for extraction of raw styrene-butadiene rubbers. A vulcanizate may be extracted with butanone, but raw rubbers and unvulcanized mixes will dissolve in this solvent.

7.1.2 Remove the extracted rubber from the apparatus and dry at 100 °C until free of solvent. Usually, a period of 1 to 2 h is required.

7.1.3 Place 0,1 to 0,2 g of the prepared test portion into the pyrolysis tube (A).

7.1.4 Introduce a small amount of the anhydrous sodium sulfate (5.4) into the collecting tube (C) to absorb any water produced during pyrolysis.

7.1.5 Place a strip of the moistened Congo red indicator paper (5.5) across the mouth of the collecting tube, as a test for halogen. (A change from red to blue indicates the presence of halogen.)

7.1.6 If alternative halogen-detecting methods are employed, use the appropriate procedure instead of that specified in 7.1.5.

7.1.7 Bring the electric furnace (D) to 550 ± 25 °C and hold at this temperature. This range is recommended to obtain fast pyrolysis, without excessive degradation and carbonization. Temperatures below the lower limit of this range should be avoided.

7.1.8 Pass a slow stream of the nitrogen (5.1) through the pyrolysis tube (A) and introduce the tube containing the prepared test portion into a hole in the aluminium block (E). [Nitrogen serves to displace air, prevent oxidation and facilitate transfer of the pyrolysis products to the collecting tube (C).] Maintain the nitrogen flow at 10 ± 2 cm³/min.

7.1.9 Continue heating to complete distillation, which may require up to 15 min.

7.1.10 Transfer a few drops of the pyrolysate from the tube to one of the polished salt plates (6.6), using a capillary pipette (6.3).

7.1.11 Place a spacer of suitable thickness (approximately 25 μm) in position, close the cell with a second plate of the same material and mount the cell in the infra-red spectrometer (6.8).

7.1.12 Record the infra-red spectrum over the wavelength range 2,5 to 15 μm , using the infra-red spectrometer (6.8).

7.1.13 Spectra shall be obtained immediately after pyrolysis to minimize further change in the pyrolysis products.

7.2 Gas flame pyrolysis (Alternative method)

7.2.1 Prepare 0,5 g of test portion as specified in 7.1.1 and 7.1.2, and place 0,1 to 0,2 g in the bottom of a small glass test-tube. Place a strip of the moistened Congo red indicator paper (5.5) across the mouth of the tube. Hold the tube horizontally and quickly bring the end containing the prepared test portion into the hot zone of a gas burner, so that the rubber is rapidly pyrolysed and charring is kept to a minimum. Note any colour change in the Congo red indicator paper, from red to blue, indicating the presence of halogen.

7.2.2 Continue heating while the pyrolysate condenses in the cool end of the tube and until distillation is substantially complete, then withdraw the test-tube from the flame. Proceed as specified in 7.1.10 to 7.1.13 inclusive.

7.2.3 As a variation of 7.2.1, the extracted test portion may be placed in a small glass test tube, the end of which has been drawn out to a retort shape. Carry out the pyrolysis as specified in 7.2.1.

WARNING — Take care to keep the delivery end of the pyrolysis tube warm enough so that pyrolysis products do not condense and block the opening, causing the tube to explode.

Collect the condensate in a small test-tube containing a small amount of the anhydrous sodium sulfate (5.4) to remove any moisture produced. Proceed as specified in 7.1.10 to 7.1.13 inclusive.

7.2.4 If alternative halogen-detecting methods are employed use the appropriate procedure instead of that specified.

8 Procedure for films cast from solution

NOTE — The methods described in 8.1 and 8.2 may give different relative peak intensities for the polymers in a given blend. The film produced by the method described in 8.2 may contain a higher proportion of the thermally less stable polymer.

8.1 Dissolution of vulcanizates

8.1.1 Prepare a test portion of approximately 2 g (or 5 g, if the presence of chloroprene rubber is suspected — see 8.1.3) as specified in 7.1.1 and 7.1.2.

8.1.2 Pyrolyse approximately 1 g of the test portion (8.1.1) and test for halogen as specified in 7.1.5 to 7.1.9 inclusive or 7.2.1 to 7.2.4 inclusive.

8.1.3 Place 1 g of the prepared test portion (8.1.1) and 50 cm³ of 1,2-dichlorobenzene (5.3.4) in a 100 cm³ flask fitted with a reflux condenser. If the presence of chloroprene rubber is suspected, prepare a test portion of approximately 5 g and place 4 g of the prepared test portion and 200 cm³ of 1,2-dichlorobenzene in a 500 cm³ flask fitted with a reflux condenser. Heat at about 120 °C until the test portion is dispersed or dissolved.

8.1.3.1 The time required for dissolution varies according to the rubber. Natural rubber requires 3 to 4 h, while chloroprene rubber may require up to 12 h. Some rubbers never dissolve completely.

8.1.3.2 Discontinue heating at the end of 12 h to minimize the risk of altering the molecular structure of the rubber.

8.1.3.3 If complete dissolution is not achieved in 12 h, sufficient rubber has usually dissolved to provide a good spectrum, although this may not be representative of both components of a blend. Partial dissolution may result in the separation of the components of a blend and further information may be obtained by pyrolysis of the dried residue.

8.1.4 Cool the mixture and transfer to a beaker containing 50 cm³ of the toluene (5.3.1).

8.1.5 If the compound does not contain carbon black, centrifuge to eliminate mineral fillers.

8.1.6 If the compound contains carbon black, add 10 to 20 g of the filter aid (6.7) and filter through filter paper.¹⁾

8.1.7 If the filtrate obtained in 8.1.6 still contains carbon black, repeat the filtration with additional filter aid.

8.1.8 Concentrate the centrifuged or filtered solution to a small volume under vacuum in a stream of the nitrogen (5.1).

8.1.9 Evaporate a few drops of the concentrated solution on a polished salt plate (6.6) to give a film thickness having 10 to 20 % transmittance at 6,9 μm.

8.1.10 Record the infra-red spectrum over the wavelength range 2,5 to 15 μm, using the infra-red spectrometer (6.8).

8.2 Mild thermal degradation for vulcanizates²⁾

8.2.1 Prepare a test portion of 2 g as specified in 7.1.1 and 7.1.2.

8.2.2 Place the prepared test portion in a test-tube capped with glass wool and heat for about 10 min in the oven (6.4), maintained at 200 ± 5 °C. Allow to cool.

8.2.3 Transfer the test portion to a beaker and add 50 cm³ of 1,1,1-trichloroethane (5.3.2). Cover with a clock-glass.

8.2.4 Allow the beaker and contents to stand for approximately 30 min on the boiling water-bath (6.5), with occasional stirring to effect dissolution of the degraded rubber.

8.2.5 Filter the mixture obtained as described in 8.2.4 through filter paper to remove any undissolved vulcanizate or fillers.

8.2.6 If free carbon is released by the vulcanizate (sometimes found with polyisoprene and butyl rubbers), add a small amount of the filter aid (6.7) to the solution before filtering.

8.2.7 Remove the solvent by evaporation, distillation or by the use of a rotary evaporator under vacuum.

8.2.8 Dissolve the residue in a small amount of the dichloromethane (5.3.3).

8.2.9 If the analyst suspects that the filtrate from 8.2.6 contains material other than rubber, which might interfere in interpretation of the final infra-red spectrum, the polymer shall be precipitated from the solution of 8.2.5 or 8.2.6 using methanol. Filter the recovered polymer and dissolve in the dichloromethane (5.3.3).

8.2.10 Evaporate a few drops of the solution in dichloromethane on a polished salt plate (6.6) to give a film thickness having 10 to 20 % transmittance at 6,9 μm.

8.2.11 Record the infra-red spectrum over the wavelength range 2,5 to 15 μm, using the infra-red spectrometer (6.8).

8.3 Dissolution of raw rubbers

8.3.1 Prepare a test portion of approximately 2 g as specified in 7.1.1 and 7.1.2.

8.3.2 Pyrolyse approximately 1 g of the prepared test portion (8.3.1) and test for halogen as specified in 7.1.5 to 7.1.9 inclusive or 7.2.1 to 7.2.4 inclusive.

1) Nitrile-butadiene rubber (NBR) could be retained on the filter paper.

2) This technique should not be used with blends suspected of containing chloroprene rubber (CR).

8.3.3 Place the remaining 1 g of the prepared test portion (8.3.1) and 50 cm³ of the dichloromethane (5.3.3) in a 100 cm³ flask fitted with a reflux condenser. Boil until the test portion is dissolved.

8.3.4 Cool and transfer to a beaker.

8.3.5 Concentrate the solution to a small volume under vacuum in a stream of the nitrogen (5.1).

8.3.6 Evaporate a few drops of the concentrated solution on a polished salt plate (6.6) to give a film thickness having 10 to 20 % transmittance at 6,9 μm.

8.3.7 Record the infra-red spectrum over the wavelength range 2,5 to 15 μm, using the infra-red spectrometer (6.8).

9 Interpretation of spectra

9.1 Reference spectra

9.1.1 Because of the different modes of presentation of spectra, it is mandatory that a set of reference spectra be prepared using the same instrument before proceeding to unknown samples.

9.1.2 Reference spectra shall be produced from test portions of known composition following the procedure specified in either clause 7 or clause 8.

NOTE — The user should compare his own reference spectra for single polymers with the reference spectra included in the annex and he should prepare further spectra for any polymers for which his spectra differ significantly from those in the annex.

9.1.3 Spectra of mixtures are not given in this International Standard because of the multiplicity of polymer combinations and proportions. Each laboratory shall prepare its own set from test portions 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 (intensity of absorption) or absorption wavelength. The experienced spectroscopist will recognize this.

9.1.5 Vulcanizates may give slightly different liquid pyrolysate and film spectra from those prepared from the corresponding raw rubber, and the interpretation of such spectra requires particular care when strong characteristic features are lacking.

9.1.6 The warnings in 9.1.4 and 9.1.5 are given to alert the spectroscopist to difficulties which may arise from the use of

this method and to emphasize the need for maintaining a reference file for films and pyrolysates of all the rubbers which may require identification.

9.1.7 In all cases, spectra shall be interpreted in the light of the test for halogen. If the test is negative, then the rubber is characterized as not being chloroprene rubber nor chloro-sulfonated polyethylene.

9.2 Tables of diagnostic absorptions

9.2.1 The tables of diagnostic absorptions (see the annex) shall be used only in conjunction with reference spectra. Their purpose is to indicate the principal absorptions by which the spectra may be related to films and pyrolysates, such as the specific nature of certain peaks, their relationship with neighbouring peaks or other regions of the spectrum, etc.

9.2.2 The tables serve to augment reference spectra by drawing attention to absorptions which are absent, thereby eliminating certain rubbers, when ambiguity could arise.

9.2.3 Absorptions are arranged in order of diagnostic value, from strong to weak. Diagnostic value means those features of the absorption, subjectively assessed, which an experienced spectroscopist recognizes as of significance in rubber identification. Such absorptions are not necessarily specific nor necessarily very intense. They are, however, associated with certain compositional or structural features of the liquid pyrolysates and films, and are reproducible in the sense that they are not seriously influenced by moderate variations in pyrolysis and dissolution conditions.

9.2.4 In general, it can be assumed with confidence that, if a particular rubber is present, apart from the exceptions stated, all the absorptions listed will appear in the spectrum of the corresponding pyrolysate or film, and, if any of these absorptions are absent, it can be inferred that the rubber in question is absent.

10 Test report

The test report shall include the following information :

- a) all details required for the complete identification of the sample;
- b) a reference to this International Standard;
- c) the method used;
- d) identification of the rubber(s) in the sample;
- e) date of test.

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Annex

Tables of diagnostic absorptions and reference spectra

A.0 Introduction

This annex gives tables of diagnostic absorptions and reference spectra for pyrolysates and films cast from solution, as indicated in the following table.

Type of rubber	Figure number	
	Raw polymer	Vulcanizate
Pyrolysates		
Isoprene rubber	2	3
Styrene-butadiene rubber	4	5
Butadiene-acrylonitrile rubber	6	7
Chloroprene rubber	8	9
Isobutene-isoprene rubber	10	11
Polybutadiene rubber	12	13
Ethylene-propylene rubber	14	15
Chlorosulfonated polyethylene	16	17
Films cast from solution		
Natural rubber	18	19
Gutta percha	20	21
Styrene-butadiene rubber	22	23
Butadiene-acrylonitrile rubber	24	25
Chloroprene rubber	26	27
Isobutene-isoprene rubber	28	29
Polybutadiene rubber	30	31
Ethylene-propylene rubber	32	33
Chlorosulfonated polyethylene	34	35

For each figure, two reference spectra are given : one with a linear wavelength scale and one with a linear wavenumber scale, representing spectra obtained using different types of machines. The arrows marked on the spectra correspond to the diagnostic absorptions given in the corresponding tables.

A.1 Pyrolysates

A.1.1 The following absorptions, when they occur, are of no diagnostic value and should not be used for rubber identification.

Wavelength	Wavenumber
μm	cm^{-1} *
3,0	3 330
3,5	2 860
5,9	1 700
6,9	1 450

* The SI unit for wavenumber is the reciprocal metre. Until such units are used on instruments and graphs, the reciprocal centimetre will be retained in this International Standard.

A.1.2 The principal absorptions in order of diagnostic value are as follows.

A.1.2.1 For isoprene rubber (see figures 2 and 3)

Wavelength	Wavenumber	Intensity
μm	cm^{-1}	
11,3	885	Very strong
7,3	1 370	Strong
12,5	800	Medium
6,1	1 640	Medium
11,0	909	Shoulder

In vulcanizates, the absorption at 12,5 μm (800 cm^{-1}) may sometimes decrease in intensity with the appearance of a broader, less intense absorption at 12,3 μm (813 cm^{-1}).

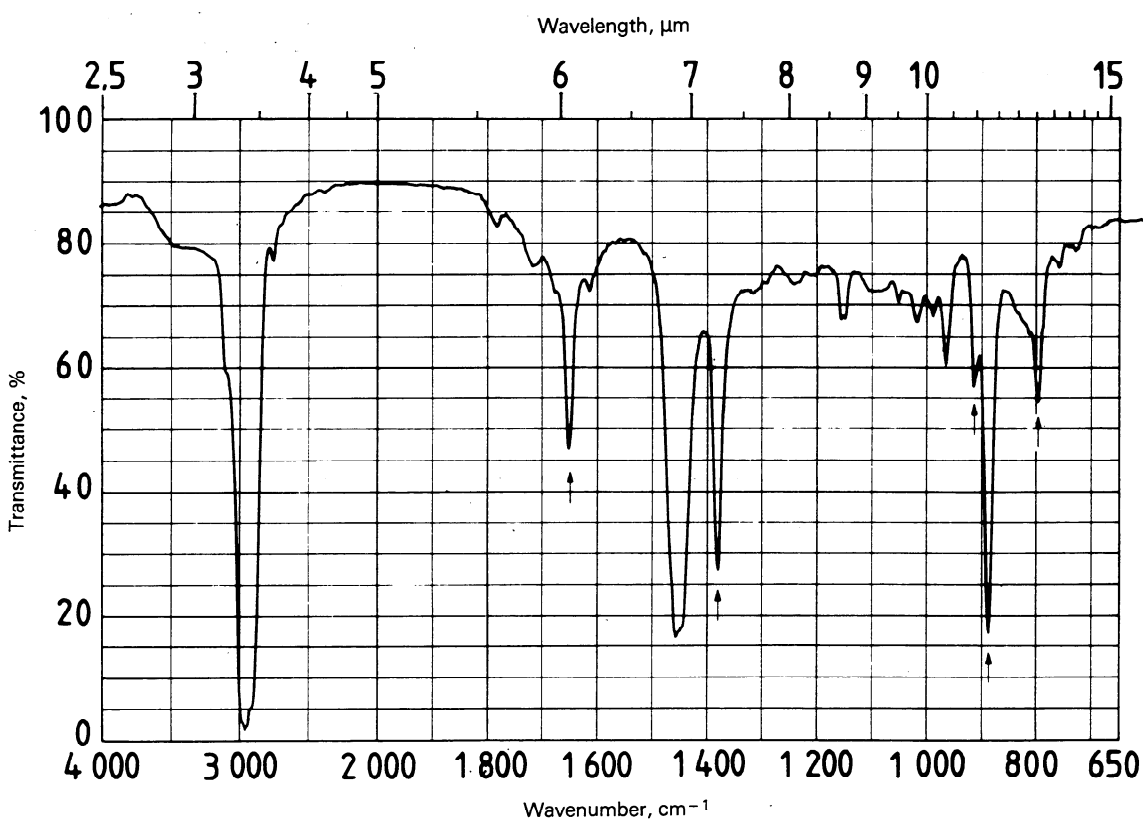
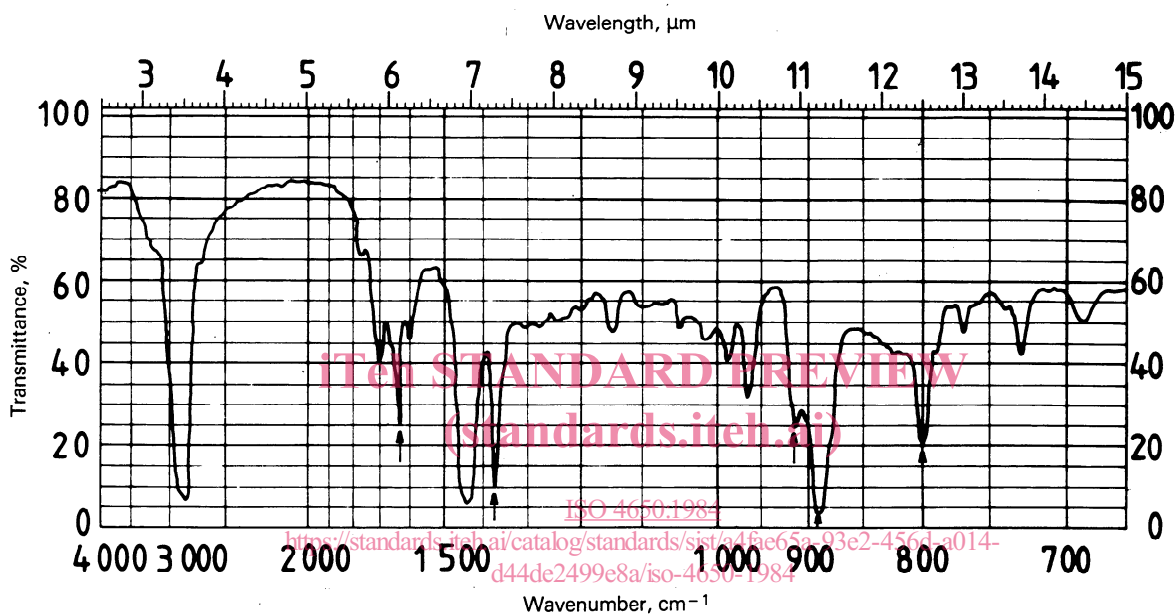


Figure 2 — Isoprene rubber — Pyrolysate of raw polymer

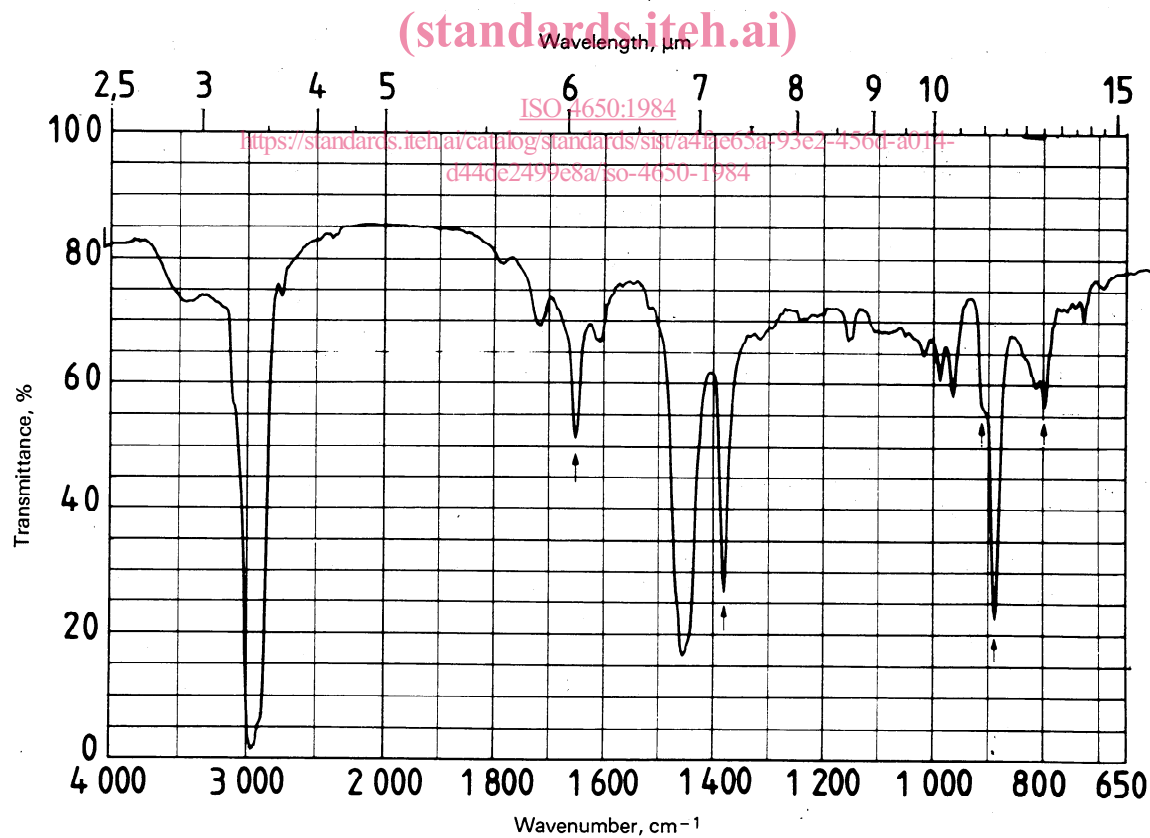
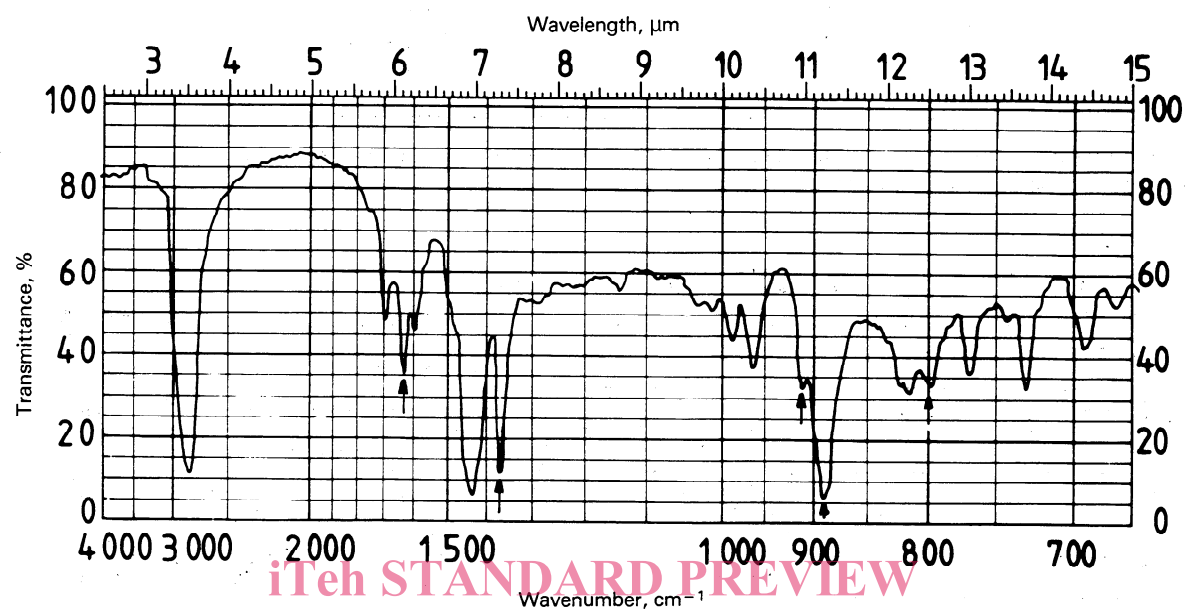


Figure 3 — Isoprene rubber — Pyrolysate of vulcanizate