



**International
Standard**

ISO 4650

**Rubber — Identification — Infrared
spectrometric methods**

*Caoutchouc — Identification — Méthodes spectrométriques dans
l'infrarouge*

**Fourth edition
2026-07**

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

This fourth edition cancels and replaces the third edition (ISO 4650:2012), which has been technically revised.

The main change is as follows:

- replacement of some of the reference spectra in [Annex B](#).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Rubber — Identification — Infrared spectrometric methods

1 Scope

This document specifies infrared spectroscopic methods using the transmission and attenuated total reflectance (ATR) methods for the identification of rubbers, including thermoplastic elastomers, either in the raw state or in the form of vulcanized or unvulcanized mixes.

The methods specified are qualitative methods only.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1407, *Rubber — Determination of solvent extract*

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Principle

4.1 General

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 document.

A comparison of the spectra resulting from 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](#).

4.2 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 infrared spectrum is recorded and then interpreted by comparison with a set of typical reference spectra.

4.3 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.

5 Types of rubber

5.1 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 [5.9](#)).

5.2 M group

- Acrylic rubber (ACM) (CAS Registry Number®¹⁾ 67254-76-6): Copolymer of ethyl acrylate (or other acrylates) and a small amount of a monomer which facilitates vulcanization.
- Copolymer of ethyl acrylate (or other acrylates) and ethylene (AEM) (CAS RN 9010-86-0).
- Chloropolyethylene (CM) (CAS RN 64754-90-1) and chlorosulfonylpolyethylene (CSM) (CAS RN 68037-39-8): The methods will not differentiate CM from CSM, and it will not differentiate between different types of CSM.
- Ethylene-propylene copolymer (EPM) (CAS RN 9010-79-1) and ethylene-propylene-diene terpolymer (EPDM) (CAS RN 25038-36-2): 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.
- Fluorocarbon rubber (FKM) (CAS RN 25190-89-0): Examination of the pyrolysate can give some information about the different grades of fluorocarbon rubber present.

5.3 O group

- Polychloromethyloxirane (CO) (CAS RN 24969-06-0): Copolymer of ethylene oxide and chloromethyloxirane (ECO) (CAS RN 24969-10-6) and terpolymers (GECO) (CAS RN 26587-37-1). Examination of the pyrolysate will not differentiate between different types of CO.

5.4 Q group

- Polydimethylsiloxane (MQ) (CAS RN 63148-62-9), polymethylphenylsiloxane (PMQ) (CAS RN 63148-58-3) and polymethyl-fluorosiloxane (FMQ): Examination of the pyrolysate will differentiate PMQ from MQ.

5.5 R group

- Butadiene rubber (BR) (CAS RN 9003-17-2): 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.
- Chloroprene rubber (CR) (CAS RN 9010-98-4): The methods will not differentiate between the different types of CR.
- Isobutene-isoprene rubber (IIR) (CAS RN 9010-85-9) and halogenated isobutene-isoprene rubbers (BIIR) (CAS RN 68441-14-5) and CIIR (CAS RN 68081-82-3) and brominated isobutylene-co-p-methylstyrene

1) CAS Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

(BIMS): Under the conditions used, it is not possible to differentiate between IIR, BIIR, CIIR and polyisobutene.

- Natural rubber (NR) (CAS RN 9006-04-6) and synthetic isoprene rubber (IR) (CAS RN 308067-72-3): 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.

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.

- Acrylonitrile-butadiene rubber (NBR) (CAS RN 9003-18-3): The methods will differentiate carboxylic acrylonitrile-butadiene rubbers (XNBRs) (CAS RN 9010-81-5) from hydrogenated acrylonitrile-butadiene rubbers (HNBRs) (CAS RN 88254-10-8). Associations of butadiene copolymers and PVC (CAS RN 25702-80-1) are included. Examination of the pyrolysate film gives some information about the acrylonitrile content.
- Styrene-butadiene rubber (SBR) (CAS RN 9003-55-8): 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.
- Polynorbornene.

5.6 T group

- Polysulfide rubbers.

5.7 U group

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

5.8 TPE group

Shall be as defined in ISO 18064.

5.9 Exceptions for blends

- Analysis of a blend of ethylene-propylene rubber with other rubbers presents difficulties when its ethylene-propylene content is below 40 %.
- The methods will not differentiate between blends of ethylene-propylene rubber with ether chlorinated polyethylene or chloro-sulfonated polyethylene, or both.
- Analysis of a blend of either natural or synthetic polyisoprene and chloroprene rubber, or both, can present difficulties, and identification of the minor component can only be possible when the content is equal to or greater than 30 % in the blend.
- 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.
- The presence of high quantities of sulfur in a vulcanizate can affect some characteristic bands.
- The methods will not differentiate NBR/PVC blends from blends of NBR with other halogenated polymers or additives.

5.10 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](#).

NOTE In spectroscopy, spectra are generally expressed as absorbance against wavenumber. Wavenumber (ν in cm⁻¹) can be converted to wavelength (λ in nm) using the following formula.

$$\lambda = \frac{10^7}{\nu}$$

6 Reagents

6.1 Nitrogen (CAS RN 7727-37-9), in pressurized cylinders, of oxygen content less than 10 mg/kg.

6.2 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).

— Methanol (CAS RN 67-56-1), of recognized analytical grade.

— Acetone (CAS RN 67-64-1), of recognized analytical grade.

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

— Chloroform (CAS RN 67-66-3), of recognized analytical grade.

— 1,2-dichlorobenzene (CAS RN 95-50-1), of recognized analytical grade.

6.4 Sodium sulfate (CAS RN 7757-82-6), anhydrous, of recognized analytical grade.

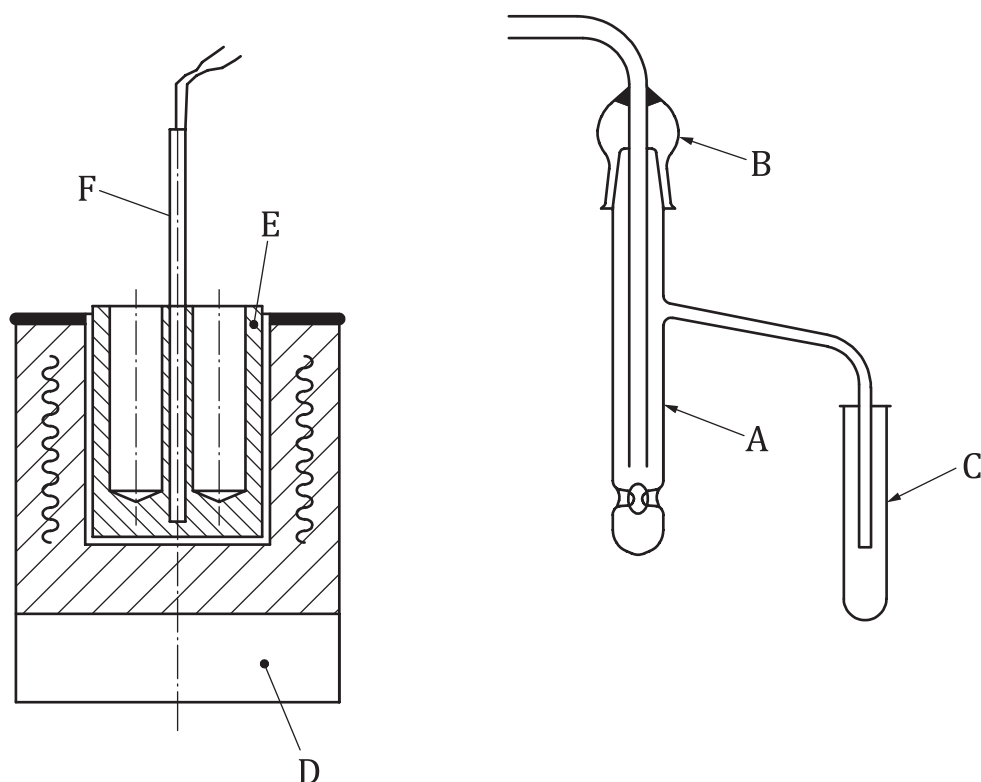
6.5 Universal pH-indicator paper.

7 Apparatus

7.1 Extraction apparatus.

The apparatus specified in ISO 1407 shall be used.

7.2 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.

**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

7.3 Capillary pipettes.

7.4 Oven, capable of being maintained at $200\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

7.5 Water bath.

7.6 Polished potassium bromide (CAS RN 7758-02-3) salt plates.

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

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

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

7.9 ATR (attenuated total reflectance) attachment with a suitable reflecting crystal, e.g. diamond (CAS RN 7782-40-3), ZnSe (CAS RN 1315-09-9), Ge (CAS RN 7440-56-4).

8 Procedure for infrared spectrometric examination using the transmission technique

8.1 Procedure for raw rubber films moulded or cast from solution

- a) Using a suitable solvent (see [6.2](#)), extract the extractable material from a test sample of 2 g to 5 g in accordance with the procedure given in ISO 1407.
- b) Dissolve a sufficient amount of the extracted rubber in a suitable solvent (see [6.3](#)), at room temperature or under reflux, to give a concentrated solution.
- c) Place a few drops of the concentrated solution on a potassium bromide salt plate ([7.6](#)) and allow the solvent to evaporate.
- d) Films of raw rubber of a suitable thickness may also be obtained by moulding.
- e) Record the spectrum from $4\,000\text{ cm}^{-1}$ to 600 cm^{-1} using the infrared spectrometer ([7.8](#)).
- f) 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.
- g) A test for halogens can be carried out as described in [8.2.2 d\)](#).

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

8.2.1 General

The methods described in [8.2.2](#) and [8.2.3](#) can give different relative absorbances for the polymers in a given blend.

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

- a) Extract a 2 g to 5 g test sample in accordance with the procedure given in ISO 1407.
- b) 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](#)).
- c) Introduce a small quantity of sodium sulfate in the collector tube C to absorb water formed in the pyrolysis.
- d) Carry out a test for halogen, for instance by placing a strip of moistened indicator paper (see [6.5](#)) 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 can cause interference. Other suitable halogen-detection methods may also be used.
- e) Bring the electric furnace D to $525\text{ °C} \pm 50\text{ °C}$ 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 recommended, however, to obtain the maximum quantity of pyrolysate for NR, IR, BR, SBR, IIR, BIIR and CIIR.
- f) After substituting the pyrolysis tube A containing prepared sample inside with nitrogen (see [6.1](#)), 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 $10\text{ cm}^3/\text{min} \pm 2\text{ cm}^3/\text{min}$.
- g) Continue the heating to complete distillation, i.e. for about 15 min.
- h) 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.

- i) Record the infrared spectrum from 4 000 cm⁻¹ to 600 cm⁻¹, performing the same checks as described in [8.1 f](#)).

8.2.3 Alternative method: Gas flame pyrolysis

Rapid pyrolysis may be performed in a test tube in place of the procedures described in [8.2.2 b](#)), [8.2.2 c](#)), [8.2.2 e](#)), [8.2.2 f](#)) and [8.2.2 g](#)).

NOTE The spectrum obtained from [8.2.3](#) cannot produce the same spectrum obtained by the preferred method in [8.2.2](#).

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

8.3.1 General

The methods described in [8.3.2](#) and [8.3.3](#) can give different relative absorbances for the polymers in a given blend. The films obtained by the method described in [8.3.3](#) can contain a higher proportion of the thermally less stable polymer.

8.3.2 Dissolution of vulcanizate

- a) Prepare a test sample of about 2 g (or 6 g if the presence of chloroprene rubber is suspected) (see [8.3.2 b](#)) and [8.3.3 c](#)) and proceed with the extraction as described in ISO 1407.
- b) Pyrolyse approximately 1 g of the prepared test sample and carry out a halogen test as described in [8.2.2 d](#)).
- c) If no chloroprene rubber is present, place 1 g of the test sample prepared in [8.3.2 a](#)) and 50 cm³ of a solvent appropriate to the rubber type (see [6.3](#)) (1,2-dichlorobenzene is suggested) in a 100 cm³ flask fitted with a reflux condenser. If chloroprene rubber is present, place approximately 5 g of the test sample prepared in [8.3.2 a](#)) with 200 cm³ of solvent in a 500 cm³ 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.

- d) If the rubber does not contain carbon black, centrifuge to eliminate mineral fillers.
- e) If the rubber contains carbon black, add 10 g to 20 g of filter aid ([7.7](#)) and filter through ordinary filter paper. Should the filtrate contain carbon black, repeat the filtration with more filter aid.

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

- f) Concentrate the centrifuged or filtered solution to a small volume under a stream of nitrogen ([6.1](#)) or reduced pressure.
- g) Evaporate a few drops of the concentrated solution on a potassium bromide salt plate.
- h) Record the infrared spectrum from 4 000 cm⁻¹ to 600 cm⁻¹, performing the same checks as described in [8.1 f](#)).

8.3.3 Mild thermal degradation of vulcanizates

- a) This technique shall not be used on blends which can contain chloroprene rubber.
- b) Prepare a test sample of 2 g as described in [8.2.2 a](#)).
- c) Place the prepared test sample in a test tube capped with glass wool and heat for about 10 min in an oven ([7.4](#)) regulated at 200 °C ± 5 °C (a temperature of 180 °C is recommended for NR, IR, BR, SBR, IIR, BIIR and CIIR rubbers).

- d) Allow the test sample to cool, transfer to a 100 cm³ flask fitted with a reflux condenser and add 50 cm³ of chloroform (6.3) to the flask. Place the flask in a hot water bath.
- e) Allow the flask and contents to remain for about 30 min in the water bath, with the solvent refluxing, to dissolve the degraded rubber.
- f) Filter the mixture obtained in 8.3.3 e) through filter paper to remove any undissolved vulcanizate and fillers. If carbon black be released from the vulcanizate, add a small amount of filter aid (7.7) to the solution before filtering.
- g) When it is suspected that the filtrate obtained in 8.3.3 f) contains material other than rubber which can interfere in the interpretation of the final spectrum, precipitate the polymer from the filtrate obtained in 8.3.3 f) using methanol. Filter off the recovered polymer and redissolve it in chloroform (6.3).
- h) Evaporate a few drops of the chloroform solution on a potassium bromide salt plate (7.6) to give a film thickness suitable for the production of an analytical spectrum.
- i) Record the spectrum from 4 000 cm⁻¹ to 600 cm⁻¹, performing the same checks as described in 8.1 f).

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

- a) 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.
- b) Clean the crystal surface before the test.
- c) Place the test sample on the crystal, ensuring good contact.
- d) Record the spectrum.

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

10 Interpretation of spectra

10.1 Reference spectra

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

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

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.

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

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

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 necessary that all the bands be examined, irrespective of their number.

10.2 Tables of diagnostic absorptions

Annex B presents tables of diagnostic absorption bands and examples of reference spectra for major rubbers as Tables B.1 to B.71 and Figures B.1 to B.71.

The tables of diagnostic absorption bands given in [Annex B](#), when used, shall be used only in conjunction with reference spectra. Their purpose is to indicate the principal absorption bands.

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

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.

11 Test report

The test report shall include the following particulars:

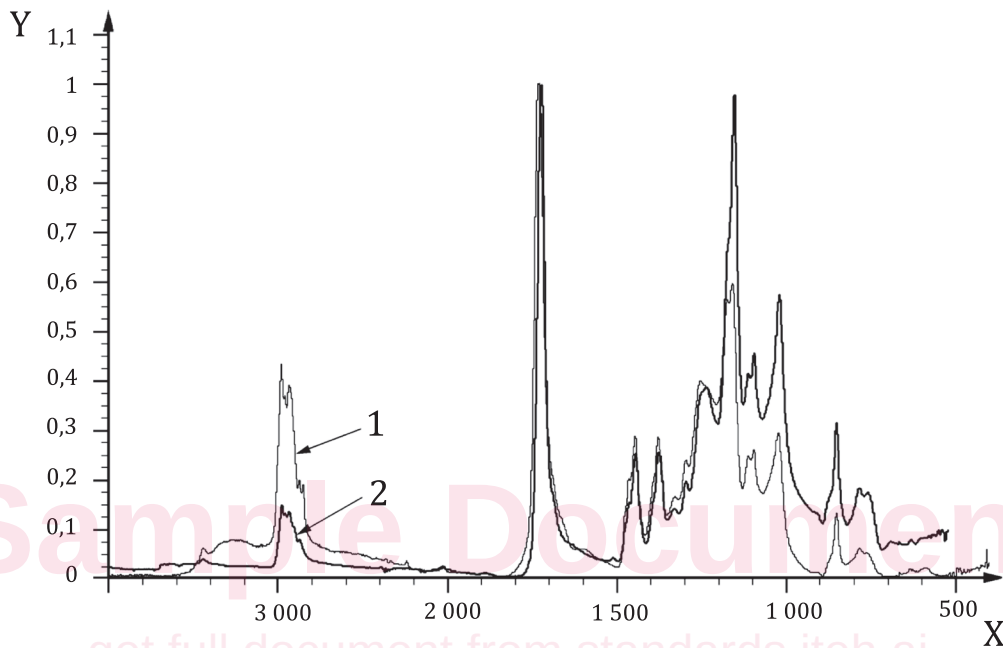
- a) sample details:
 - 1) a full description of the sample;
 - 2) if appropriate, the method of preparation of the test sample from the sample;
- b) test method:
 - 1) a reference to this document and its year of publication, i.e. ISO 4650:2026;
 - 2) the test procedure used (film or ATR);
- c) details of any procedures not specified in this document;
- d) the test results, i.e. identification of the rubber(s) in the sample;
- e) the date of the test.

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

Annex B (informative)

Absorption characteristics and reference spectra

B.1 General

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

Comparisons between sample spectra and reference spectra must take into account the position of the bands, how many there are, their relative intensity and their shape.

All the bands in a spectrum shall 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

For ease of use, the scale chosen for the presentation of the spectra is designed to clearly show the specific absorption bands.

[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					