

Elastomeric parts for parenterals and for devices for pharmaceutical use —

Part 2: Identification and characterization

ICS 11.040.20

National foreword

This British Standard is the UK implementation of EN ISO 8871-2:2004+A1:2014. It is identical to ISO 8871-2:2003, incorporating amendment 1:2005. It supersedes BS EN ISO 8871-2:2004, which is withdrawn.

The start and finish of text introduced or altered by amendment is indicated in the text by tags. Tags indicating changes to ISO text carry the number of the ISO amendment. For example, text altered by ISO amendment 1 is indicated by A1 A1 .

The UK participation in its preparation was entrusted to Technical Committee CH/212, IVDs.

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

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ICS 11.040.20

English version

Elastomeric parts for parenterals and for devices for
pharmaceutical use - Part 2: Identification and characterization
(ISO 8871-2:2003)

Eléments en élastomère pour administration parentérale et
dispositifs à usage pharmaceutique - Partie 2: Identification
et caractérisation (ISO 8871-2:2003)

This European Standard was approved by CEN on 15 July 2004.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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Foreword

The text of ISO 8871-2:2003 has been prepared by Technical Committee ISO/TC 76 "Transfusion, infusion and injection equipment for medical and pharmaceutical use" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 8871-2:2004 by CMC.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2005, and conflicting national standards shall be withdrawn at the latest by March 2005.

This document supersedes EN ISO 8871:1997.

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The text of ISO 8871-2:2003 has been approved by CEN as EN ISO 8871-2:2004 without any modifications.

Foreword to amendment A1

This document (EN ISO 8871-2:2004/A1:2014) has been prepared by Technical Committee ISO/TC 76 "Transfusion, infusion and injection, and blood processing equipment for medical and pharmaceutical use" in collaboration with Technical Committee CEN/TC 205 "Non-active medical devices" the secretariat of which is held by DIN.

This Amendment to the European Standard EN ISO 8871-2:2004 shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2014, and conflicting national standards shall be withdrawn at the latest by December 2014.

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The text of ISO 8871-2:2003/Amd 1:2005: has been approved by CEN as EN ISO 8871-2:2005/A1:2014 without any modification.

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**Elastomeric parts for parenterals and for
devices for pharmaceutical use —**

**Part 2:
Identification and characterization**

*Éléments en élastomère pour administration parentérale et dispositifs à
usage pharmaceutique —*

Partie 2: Identification et caractérisation



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Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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 8871-2 was prepared by Technical Committee ISO/TC 76, *Transfusion, infusion and injection equipment for medical and pharmaceutical use*.

Together with the other parts (see below), this part of ISO 8871 cancels and replaces ISO 8871:1990, which has been technically revised.

ISO 8871 consists of the following parts, under the general title *Elastomeric parts for parenterals and for devices for pharmaceutical use*:

- *Part 1: Extractables in aqueous autoclavates*
- *Part 2: Identification and characterization*
- *Part 3: Determination of released-particle count*
- *Part 4: Biological requirements and test methods*
- *Part 5: Functional requirements and testing*

Introduction

The elastomeric parts specified in the various parts of this International Standard are produced from a material which is usually called “rubber”. However, rubber is not a unique entity, since the composition of rubber materials may vary considerably. The base elastomer and the type of vulcanization have a major influence on the principle characteristics of an individual rubber material, as do additives such as fillers, softeners and pigments. These may have a significant effect on the overall properties. The effectiveness, purity, stability and safe handling of a drug preparation may be affected adversely during manufacture, storage and administration if the rubber part used has not been properly selected and validated (approved).

Elastomeric parts for parenterals and for devices for pharmaceutical use —

Part 2: Identification and characterization

1 Scope

This part of ISO 8871 specifies evaluation procedures applicable to elastomeric parts used for drug containers and medical devices in order to guarantee the product identity between the samples evaluated in the (suitability test) acceptance process and the current supplies. The physical and chemical test procedures specified in this part of ISO 8871 permit the determination of the typical characteristics of rubber materials, and may serve as a basis for agreements between manufacturer and user regarding the product consistency in subsequent supplies. An appropriate set of tests is selected, depending upon the type of rubber and its application.

This part of ISO 8871 does not specify other requirements for rubber materials. These are laid down in the relevant product standards.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 48:1994, *Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 247:1990, *Rubber — Determination of ash*

ISO 2781:1988, *Rubber, vulcanized — Determination of density*

ISO 8871-1:2003, *Elastomeric parts for parenterals and for devices for pharmaceutical use — Part 1: Extractables in aqueous autoclavates*

3 Tests

3.1 General

Rubber is a complex material and not generally definable. The only property which all elastomeric materials have in common is a special type of resilience or elasticity. When a strip of rubber is stretched, it will extend by up to many times its original length without breaking. On release of the stretching force, it snaps back to its original size and shape virtually unaltered. Similarly, one can squeeze it, twist it or distort it in any direction comparatively easily, and it will spring back again to its original shape unchanged.

Owing to its three-dimensional network, achieved by chemical cross-linking of the polymer chains during vulcanization, rubber is practically insoluble in solvents such as tetrahydrofuran, although considerable reversible swelling may occur; this characteristic differentiates rubber from pseudo-elastic materials, such as poly(vinyl chloride) and certain thermoplastic elastomers.

In view of the complexity of rubber, the identity of a given elastomeric material cannot be verified just by applying a single physical or chemical test, and a set of tests is needed for reliable identification.

The manufacturer shall guarantee that all elastomeric parts of current supplies have been produced from the same formulation and that they exhibit the same characteristics as the samples which have been given to the user first and the suitability of which has been proved.

3.2 Hardness

Hardness shall be determined in accordance with ISO 48.

3.3 Density

Density shall be determined in accordance with the procedure described in ISO 2781:1988, method A.

3.4 Ash

The inorganic residue after combustion shall be determined as described in ISO 247:1990, method A.

A1 3.5 Infrared spectrum

A very simple method to create a fingerprint of a rubber material is to record an infrared (IR) spectrum. The two common methods for obtaining an IR spectrum of a rubber material are pyrolysis IR and surface IR/ATR (attenuated total reflectance)-technique.

The pyrolysis IR can be obtained as described in Annex A. The surface IR/ATR can be obtained as described in Annex H. The spectra should be compared to a spectrum obtained by the same IR-method on a reference sample of the material.

In practice, pyrolysis IR requires a time-consuming sample preparation and in addition needs the cautious handling of hazardous vapours and oils.

In contrast to this, the surface IR/ATR offers the possibility to obtain a fingerprint from an elastomeric part with minimum or no sample preparation. A1

3.6 Compression set

The compression set indicates the degree of permanent deformation remaining after compression at a constant deformation and defined temperature for a defined time. The compression set shall be determined in accordance with Annex B.

3.7 Swelling

Elastomeric materials are subject to varying degrees of swelling when exposed to organic solvents; the degree of volume and/or mass increase is primarily influenced by the type of elastomer. Swelling requires special care when the rubber components are in contact with emulsions or oily vehicles.

The relevant procedure is specified in Annex C.

3.8 Development of a fingerprint by gas chromatography

The elastomeric materials under examination are extracted in a solvent, which does not dissolve but might swell the rubber. The extract is injected into a gas chromatograph. The chromatogram obtained exhibits a typical profile and can be used as a fingerprint for identification purposes. Furthermore, GC-coupling techniques, e.g. GC-MS, may provide additional information about the composition of the extract.

The relevant procedure is specified in Annex D.

3.9 Detection of volatile substances by gas chromatography

Elastomeric materials may release volatile substances. These may originate from one of the following categories of material:

- oligomers or process aids present in the base polymer;
- stabilizers or antioxidants;
- softeners.

The relevant procedure is specified in Annex E.

3.10 Determination of residual moisture

During treatments typical for the pharmaceutical industry, elastomeric parts can absorb moisture in considerable quantities. During storage of the drug unit, the trapped moisture may be released and absorbed by the drug product, thus reducing the effectiveness of the drug (critical case: lyophilized drugs). The nature of the absorption and desorption processes is affected by the composition of the rubber, the type of treatment (e.g. steam autoclaving) and the efficiency of any subsequent drying process.

The relevant procedure is specified in Annex F.

3.11 Determination of fingerprint by thermogravimetric analysis (TGA)

Elastomeric parts are composed of components which can be classified relative to their performance under thermal treatment, as follows:

- base polymers;
- inorganic fillers;
- substances volatile at elevated temperatures;
- carbon black.

The relevant procedure is specified in Annex G.

3.12 Determination of extractables in aqueous autoclavates

Elastomeric materials may release substances of undetermined nature in water. For the general assessment of the chemical cleanliness of closures, the determination of overall parameters such as oxidizable materials and electrical conductivity can be used.

The relevant test procedures are specified in ISO 8871-1.

4 Preparation of samples for testing

4.1 Treatment before testing

Since the various test procedures may require different pretreatments, such treatment is specified in each annex.

It is generally assumed that samples of rubber parts will be provided in a clean state in accordance with the state of the art. In order to avoid recontamination, they shall be contained in protective packaging. Any particular treatment or method of packaging to be carried out by the manufacturer shall be subject to agreement between the manufacturer and the customer.

4.2 Number of samples needed for the tests

Due to the large number of tests in this part of ISO 8871 and their complexity, usually not all of the tests are performed in each investigation. For this reason, the number of samples needed shall be agreed on between the manufacturer and the test laboratory. Each annex specifies the number of samples which are necessary to perform that specific test.

5 Reagents and materials

5.1 Use only reagents of recognized analytical grade and purified water prepared by distillation or by other suitable means. The conductivity of the water used shall not exceed 3,0 $\mu\text{S}/\text{cm}$.

NOTE Purified water as specified by various national pharmacopoeias corresponds to grade 1 or 2 of ISO 3696.

5.2 All glass equipment shall be made from borosilicate glass.

Annex A (informative)

Identification of elastomeric material by pyrolysis IR

A.1 General

When rubber parts are exposed to dry heat with restricted access to oxygen, the elastomeric matrix is thermally disintegrated and the rubber is converted into polymer fragments which appear in the form of vapour or oils of various viscosities.

These oily products are used to produce an IR spectrum which can serve to identify the original rubber material.

A.2 Reagents and materials

A.2.1 Dry, filtered acetone, to clean the KBr discs.

A.2.2 Indicator paper.

A.2.3 Copper wire.

A.2.4 Acetone.

A.2.5 Trichloromethane.

A.2.6 Sodium sulfate, anhydrous.

A.3 Apparatus

A.3.1 IR spectrometer, to produce IR spectra in the range from 400 cm^{-1} to $4\,000\text{ cm}^{-1}$ and from 0 % to 100 % transmission.

A.3.2 Potassium bromide (KBr) discs, including spacers and clamps.

A.3.3 Desiccator.

A.3.4 Bunsen burner.

A.3.5 Test tubes, for the pyrolysis process.

A.3.6 Soxhlet extraction apparatus (optional).

A.4 Sample preparation

Cut about 3 g of a rubber part into pieces of about $3\text{ mm} \times 3\text{ mm}$.

Optionally, extract the rubber pellets thus produced with acetone in a Soxhlet extractor under reflux for 8 h.

A.5 Pyrolysis

Place 0,2 g to 2 g of the rubber pellets in a test tube. Heat with a bunsen burner, carefully driving off any water which may initially condense above the sample. Subsequently, with the test tube in a nearly vertical position, expose the rubber pellets to the blue area of the flame. The pyrolysis products condense as an oil in the cool zone of the test tube. During the pyrolysis, the pH of the vapour released may be checked with wet indicator paper. An acidic reaction indicates the presence of halogen in the rubber [parallel to this pH check, a Beilstein (copper wire) test can be performed].

The reproducibility of the procedure specified above is good enough to obtain spectra for qualitative interpretation. The pyrolysis does not have to be performed under nitrogen at constant temperature.

A.6 Recording the spectrum

A.6.1 The liquid pyrolysis product obtained as described in Clause A.5 is best dried while still in the test tube over Na_2SO_4 in a desiccator. Then place it between two clean polished KBr discs. Usually, a thin layer (up to 0,03 mm) is sufficient to give a transmission spectrum in the range from 0 % to 80 %. If necessary, produce a thicker layer of pyrolysed material by using suitable spacers. The pyrolysed material should be evenly distributed without any included air bubbles.

A.6.2 Record the spectrum as a transmission spectrum ranging from 400 cm^{-1} to $4\,000\text{ cm}^{-1}$.

A.7 Interpretation

A.7.1 General

The pyrolysis process is a very complex one, which means that no two spectra produced will be identical, even if the sample preparation is the same. For this reason, particular features of the spectrum which are typical of the type of rubber are selected for identification purposes. Since the influence of oxygen during the pyrolysis cannot be excluded, those features of the spectrum produced by oxidation products (alcohols, ethers, aldehydes and acids) are disregarded.

A.7.2 Expression of results

Taking into consideration the limitations set out in A.7.1, compare the spectrum obtained with reference spectra at the wavelengths characteristic of the features of interest.

Record the result as the spectrum obtained, together with the results of the analysis of the spectrum.

Annex B (informative)

Determination of compression set

B.1 General

Determination of the compression set provides information about the performance of elastomeric materials when exposed to constant deformation over an extended time at room temperature or at elevated temperature. For details, see ISO 815.

B.2 Apparatus

B.2.1 Compression apparatus, consisting of two or more parallel, flat, highly polished chromium-plated steel or stainless-steel plates between the faces of which the test pieces are compressed. The surface roughness R_a of the compression plates shall not exceed $0,4 \mu\text{m}$. The plates shall be sufficiently rigid to withstand the stress produced without bending, and of sufficient size to ensure that all the compressed test pieces remain within the area of the plates. The plates shall be held together by bolts of suitable size. Steel spacers, preferably in the form of rings, of thickness between $4,7 \text{ mm}$ and $4,8 \text{ mm}$, shall be used to ensure the required compression. The spacers shall be of such a width that contact with the compressed test pieces is avoided. While the spacer thickness may be between $4,7 \text{ mm}$ and $4,8 \text{ mm}$, the thickness of the spacers used in a series of related tests shall not vary by more than $0,01 \text{ mm}$.

B.2.2 Oven: Any well-designed, uniformly heated air oven capable of maintaining the compression apparatus and test pieces at the required temperature within the tolerance limits given in Clause B.4 is suitable.

B.3 Samples

As the test cannot be performed on the closures themselves, use type B test pieces [discs with a diameter of $(13 \pm 0,5) \text{ mm}$ and a thickness of $(6,3 \pm 0,3) \text{ mm}$] prepared as specified in ISO 815.

B.4 Test conditions

Carry out the test under the following conditions (selected from those in ISO 815):

- compression $\approx 25 \%$
- duration of test $24 \frac{0}{-2} \text{ h}$
- temperature $(70 \pm 1) ^\circ\text{C}$

B.5 Procedure

B.5.1 The compression apparatus shall be at standard laboratory temperature and its operating surfaces carefully cleaned before use. If a lubricant is applied, it shall consist of a thin coating of a lubricant having substantially no action on rubber. For most purposes, a silicone or fluorosilicone fluid is suitable.

Lubrication of the operating surfaces of the compression apparatus is optional. While giving more reproducible results, lubrication may somewhat alter the compression set values. If a lubricant is not employed, the test piece surfaces shall be free from mould lubricants or dusting powder.

B.5.2 Measure the thickness of each test piece.

B.5.3 Place the test pieces between the pairs of plates together with the necessary spacers. Tighten the screws so that the plates are drawn together uniformly until they are in contact with the spacers while the rubber pieces are squeezed. The use of test pieces with a thickness of $(6,3 \pm 0,3)$ mm in combination with spacers of thickness between 4,7 mm and 4,8 mm ensures that the compression applied to the rubber discs is approximately 25 % of the initial thickness of the test pieces.

B.5.4 Without delay, introduce the compression apparatus containing the test pieces into the central part of the oven which is operating at (70 ± 1) °C. After 24 h, remove the apparatus from the oven, loosen the bolts and transfer the test pieces quickly to a wooden surface. Leave them to recover at the standard laboratory temperature for (30 ± 3) min, then measure their thickness.

B.5.5 Cut the test pieces along two diametrical lines. If any internal defects such as gas bubbles are found, discard the test piece.

B.6 Expression of results

The compression set C , expressed as a percentage of the initial compression, is given by the following equation:

$$C = \frac{h_0 - h_1}{h_0 - h_s} \times 100$$

where

h_0 is the initial thickness, in millimetres, of the test piece;

h_1 is the thickness, in millimetres, of the test piece after recovery;

h_s is the height, in millimetres, of the spacer.

Report the mean value and the standard deviation.

The results for the three test pieces shall not deviate by more than 10 % from the mean compression.

Annex C (informative)

Swelling behaviour in oils

C.1 General

The rubber parts to be examined are stored in a mixture of benzyl alcohol and peanut oil for 24 h at a temperature of (70 ± 2) °C.

After exposure to the oil, the changes in mass and volume are determined.

C.2 Apparatus and materials

C.2.1 100 ml conical flasks, with ground-glass stoppers.

C.2.2 Drying oven.

C.2.3 Ethanol (96 %).

C.2.4 Benzyl alcohol.

C.2.5 Peanut oil.

C.3 Procedure

Weigh each of five closures in air and in a suitable liquid (ethanol, or water with a tiny amount of detergent). Place each test piece in a 100 ml conical flask, add a mixture of 5 ml benzyl alcohol and 45 ml peanut oil and stopper the flask. Store for 24 h at (70 ± 2) °C in the oven.

At the end of the exposure period, remove the test pieces from the oil, clean them with ethanol and allow them to dry at room temperature. Reweigh the test pieces in air and in the liquid used for the initial weighing.

C.4 Determination of increase in mass

Calculate the increase in mass $\Delta m/m_0$ of each test piece, as a percentage, as follows:

$$\frac{\Delta m}{m_0} = \frac{m - m_0}{m_0} \times 100$$

where

m_0 is the mass of the test piece before swelling;

m is the mass of the test piece after swelling.

C.5 Determination of increase in volume

Calculate the increase in volume $\Delta V/V_0$ of each test piece, as a percentage, as follows:

$$\frac{\Delta V}{V_0} = \frac{(m - m_F) - (m_0 - m_{F0})}{m_0 - m_{F0}} \times 100$$

where

m_0 is the mass in air of the test piece before swelling, in grams;

m_{F0} is the apparent mass in the immersion liquid of the test piece before swelling, in grams;

m is the mass in air of the test piece after swelling, in grams;

m_F is the apparent mass in the immersion liquid of the test piece after swelling, in grams.

Annex D (informative)

Development of a fingerprint by gas chromatography

D.1 General

Rubber parts are extracted with *n*-heptane. Substances migrating from the rubber matrix into the *n*-heptane are analysed by gas chromatography. The gas chromatogram obtained is evaluated as a fingerprint, including a semi-quantitative assessment. The chromatogram is not used to obtain information on individual components of the rubber.

D.2 Apparatus and materials

D.2.1 *n*-Heptane.

D.2.2 Gas chromatograph, suitable for operation with a capillary column, with a split/splitless injector and a flame-ionization detector (FID).

D.2.3 Capillary column, length 50 m, inner diameter 0,32 mm, stationary phase OV 1701 or similar, film thickness 0,2 µm.

D.2.4 Carrier gas: nitrogen, 99,999 % pure.

D.2.5 Rotary shaker.

D.2.6 50 ml conical flask, with ground-glass stopper.

D.3 Preparation of test solution

Immerse rubber parts having a total surface area of 30 cm² in 20 ml of *n*-heptane in a conical flask. Agitate the suspension in a shaker for 1 h at room temperature (speed: 180 min⁻¹).

Filter the solution obtained through a disposable 0,2 µm filter. The test solution has limited stability. Prepare the solution and perform the GC analysis on the same day therefore.

D.4 GC analysis

Carry out the analysis as indicated in Tables 1 and 2. The following are typical conditions:

- Column pressure: 55,2 kPa (8,0 psi)
- Septum flush: 2,5 ml/min
- Injector temperature: 250 °C to 270 °C
- Injection volume: 1 µl (use of automatic injector with an injection volume of 5 µl also recommended)
- Detector temperature: 280 °C

Table D.1 — Separation programme

Time min	Split ml/min
0	0
0,5	20

Table D.2 — Column temperature

Time min	Heating rate °C/min	Temperature °C
0	—	120
3	—	120
—	6	—
23	—	240
—	15	—
24,3	—	260
44,3	—	260

Re-equilibration time after cooling to 120 °C: 5 min.

If it becomes necessary to dilute the test solution, the degree of dilution shall be reported.

Using a mass spectrometer as the detector may provide additional information about the nature of the components released by the rubber under the conditions described. In this case, the carrier gas may be changed and the instrument parameters adjusted appropriately.

D.5 Expression of results

Record the result as the chromatogram obtained, together with the results of the analysis of the chromatogram.

Annex E (informative)

Analysis of volatile components by headspace gas chromatography

E.1 General

Rubber parts are heated in a glass container closed with a PTFE-coated septum. A defined amount of the headspace gas is then removed by syringe and analysed by gas chromatography.

E.2 Apparatus and materials

E.2.1 Gas chromatograph, equipped for operation with a capillary column and a flame-ionization detector (FID).

E.2.2 Capillary column, length 25 m, inner diameter 0,32 mm, stationary phase SE 54 (phenyl/methyl polysiloxane) or similar, thickness 1 μm .

E.2.3 Carrier gas: nitrogen.

E.2.4 Block heater, adjustable, fitted with a thermostat.

E.2.5 Gastight syringe, volume 1 ml, or a commercially available automatic **headspace sample injector**.

E.3 Procedure

E.3.1 Cut about 5 g of rubber parts into pieces approximately 0,5 cm \times 0,5 cm in size and place them into a 20 ml glass container, closed with a laminated polytetrafluoroethylene (PTFE) septum.

E.3.2 Heat the glass container in the block heater for 1 h at a temperature of 115 °C. Subsequently, withdraw 1 ml of the headspace gas with a gastight 1 ml syringe preheated to \approx 120 °C, and immediately inject into the gas chromatograph.

E.3.3 Carry out the analysis as indicated in Table 3. The following are typical conditions:

- column pressure: 0,6 bar
- injection volume: 1 ml
- injector temperature: 250 °C
- detector temperature: 280 °C
- septum flush: 2 ml/min
- split (0 min): 15 ml/min to 20 ml/min

Table E.1 — Column temperature

Time min	Heating rate °C/min	Temperature °C
0	—	50
4	—	50
—	5	—
14	—	100
—	20	—
21	—	240
31	—	240

Using a mass spectrometer as the detector may provide additional information about the nature of the components released by the rubber under the conditions described. In this case, the carrier gas may be changed and the instrument parameters adjusted appropriately.

E.4 Expression of results

Record the result as the chromatogram obtained, together with the results of the analysis of the chromatogram.

Annex F (informative)

Determination of residual moisture

F.1 General

Rubber closures are usually sterilized in a steam autoclave, and dried afterwards by various methods. The water content near the surface of the closure can be determined, after autoclaving or drying, by means of a coulometric (Karl Fischer) method. This is mainly of interest for closures intended to be used for freeze-dried products (see ISO 8362-5 and ISO 8536-6) or other dry products.

The determination can provide detailed information about the behaviour of rubber material with respect to moisture by measuring the moisture level at different stages in the sterilization and drying processes.

F.2 Pretreatment of samples

F.2.1 No pretreatment

The closures are taken out of their package (e.g. carton) and tested without any pretreatment.

F.2.2 Steam-autoclaving pretreatment

Place at least 10 closures, as delivered, in a Petri dish. Treat the closures in a steam autoclave for 30 min at (121 ± 2) °C. Before determining the residual moisture, remove the adhering water using a soft tissue.

F.2.3 Steam-autoclaving and drying pretreatment

Autoclave a sufficiently large number of closures as described in F.2.2. Place the autoclaved closures on a Petri dish with the flange side down and dry them in steps in an oven at a temperature of 110 °C.

Remove at least 10 closures at 3 h, 5 h, 7 h and 15 h intervals and allow them to cool before testing.

F.3 Determination of residual moisture

Carry out sample preparation and determine the residual moisture in accordance with ISO 8362-5:1995, Annex D, or ISO 8536-6:1995, Annex E.

Annex G (informative)

Determination of a fingerprint by thermal gravimetry (TG)

G.1 General

Elastomeric parts are heated under defined conditions in a defined atmosphere (e.g. inert gas or air). The change in mass as a function of temperature and oven atmosphere is recorded.

In an oxidizing atmosphere, an increase in mass may be observed, depending on the fillers present. The TG curve obtained permits qualitative and quantitative determination of the components of the material (TG step analysis).

The method is not suitable for compounds containing mineral fillers, such as carbonates or hydrated aluminium oxides, which decompose at temperatures approaching 650 °C or 850 °C. When these temperatures are used, suitable corrections based on prior knowledge of filler behaviour at elevated temperatures shall be employed.

G.2 Apparatus

As specified in ISO 9924-1:2000, Clause 5, the thermogravimetric balance accurate to at least 0,1 µg.

G.3 Reagents

As specified in ISO 9924-1:2000, Clause 4.

G.4 Preparation of samples

Cut a sample of approximately 10 mg into three to five small pieces (to increase the surface area) and distribute the pieces evenly in a crucible.

NOTE It is important that the surface area of the sample is large enough for it not to restrict the rate of change in mass on heating.

G.5 Procedure

Place the sample in the thermogravimetric balance, checking that the pieces of sample are still evenly distributed in the bottom of the crucible. Set the oven temperature and the atmosphere programmer so that the programme will clearly differentiate between the various components of the elastomer.

The following typical programme¹⁾ is suitable for many elastomeric materials.

Beginning after measurement of the tare mass of the crucible, carry out the following steps:

Step 1: Hold at 30 °C for 0,1 min without purge gas (for automatic measurement of the sample mass).

1) This programme is a compromise between accuracy of measurement and the time needed to complete the run.

Step 2: Heat from 30 °C to 650 °C at 30 °C/min, purging with nitrogen at 100 ml/min.

Step 3: Heat from 650 °C to 1 000 °C at 30 °C/min, purging with air at 100 ml/min.

G.6 Quantitative assessment

A TG curve represents the change in mass of the sample as a function of temperature or time.

Apply a buoyancy correction to the curve.

Produce a differential curve.

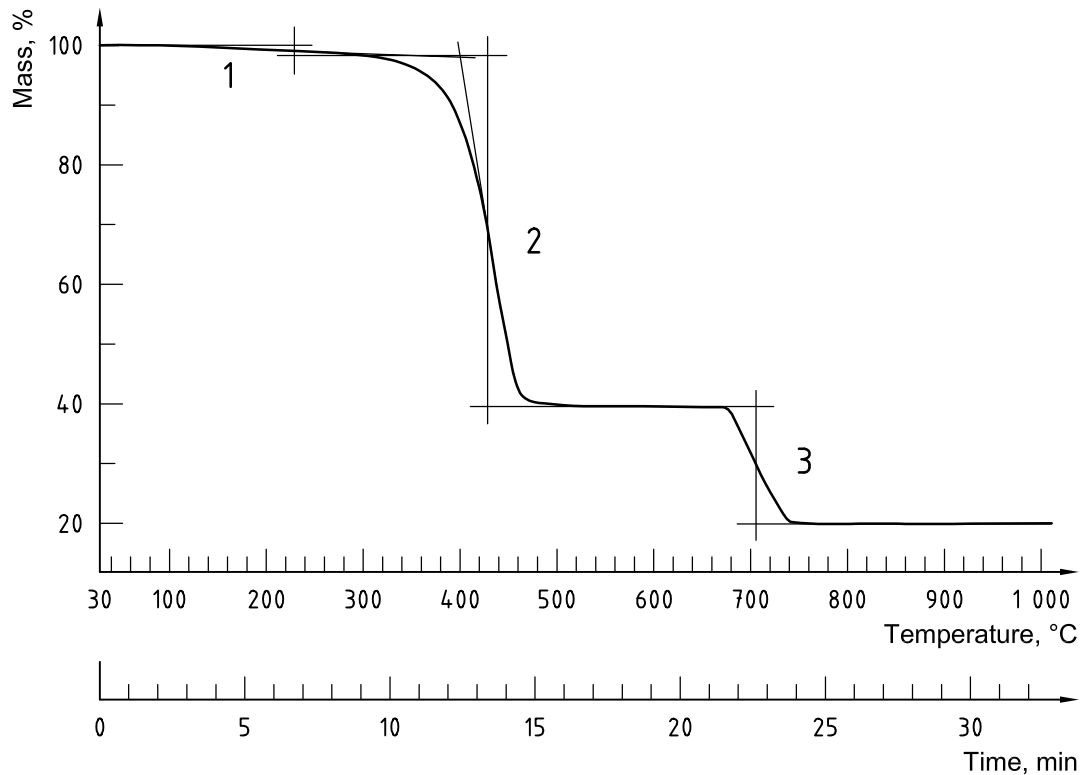
Analyse the two curves in the usual way. The heights of the steps give information about the quantitative composition of the sample, while the temperatures of the steps provide information about the type of elastomer.

G.7 Expression of results

Record the result as the TG curves obtained, together with the results of the analysis of the curves. The necessary calculations are generally made by a software programme.

G.8 Test report

As specified in ISO 9924-1:2000, Clause 9.



Key

1	removal of volatile matter	- 1,765 1 % (- 0,202 4 mg)
2	pyrolysis of elastomer	- 58,881 8 % (- 6,752 6 mg)
	point of inflection	435,19 °C
	extrapolated onset temperature	396,92 °C
	slope	- 0,50 %/s
3	combustion of carbon black	- 19,342 2 % (- 2,218 2 mg)
	residue	20,012 6 % (2,295 0 mg) (white crumb)
	point of inflection	704,40 °C

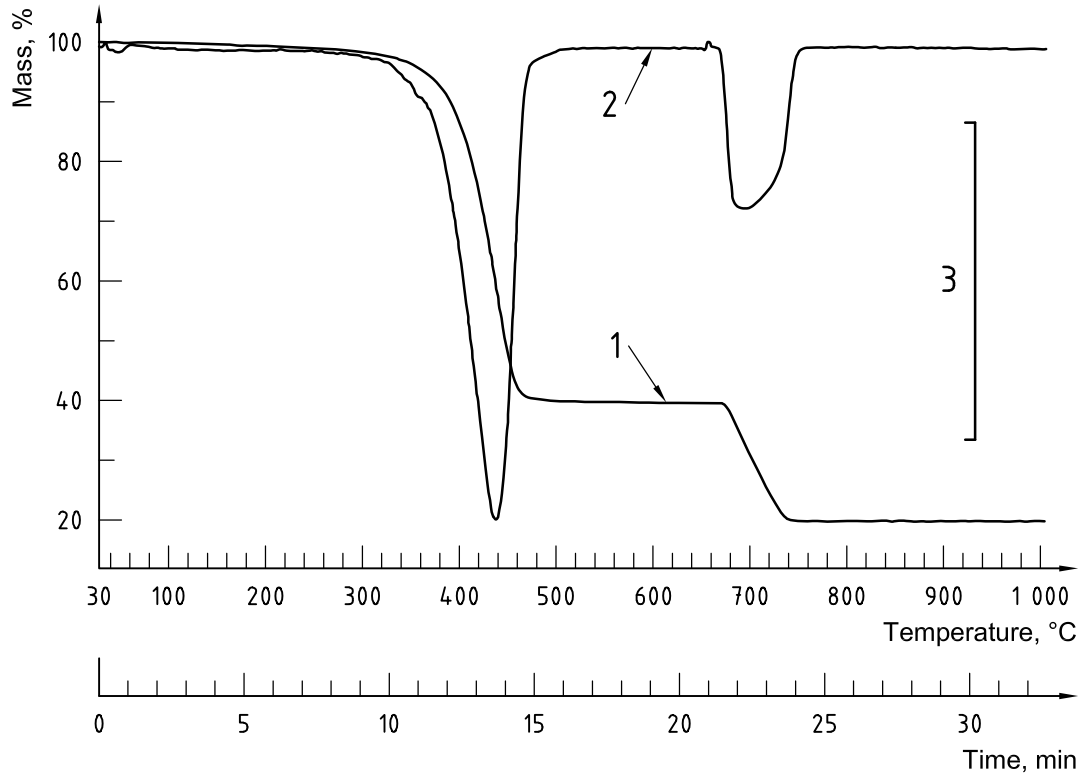
Temperature programme:

30 °C for 0,1 min

30 °C to 650 °C at 30 °C/min (purge gas N₂ at 100 ml/min)

650 °C to 1 000 °C at 30 °C/min (purge gas air at 100 ml/min)

Figure G.1 — Typical TG curve for a carbon-black-filled bromobutyl elastomer



Key

- 1 TG curve
- 2 differential TG curve
- 3 ordinate scale for the differential TG curve (2) = %/min

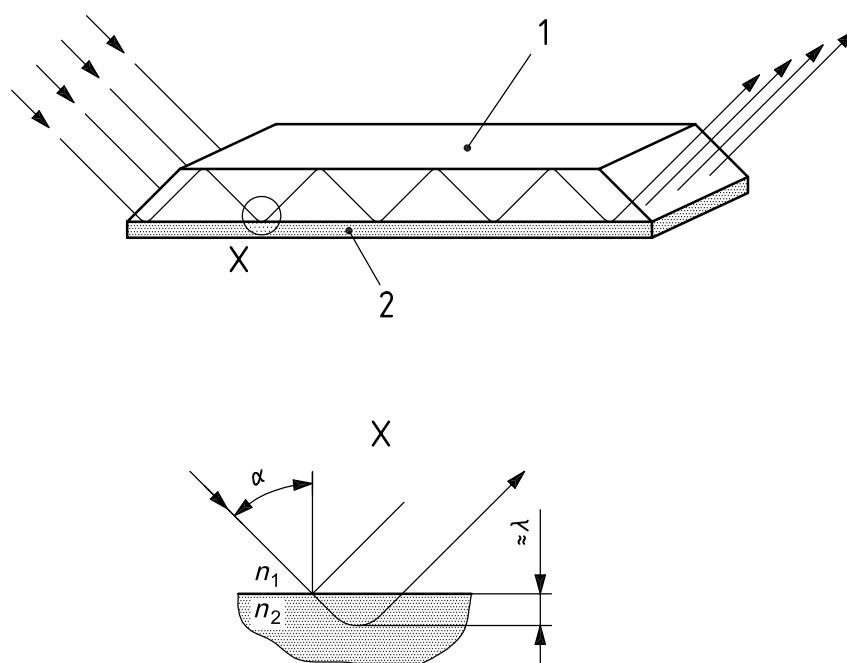
Figure G.2 — Typical TG curve, with superimposed differential curve, for a carbon-black-filled bromobutyl elastomer

Annex H
(informative)

**Determination of a fingerprint by surface infrared spectroscopy
(ATR, attenuated total reflection)**

H.1 General

The principle of ATR measurement is shown in Figure H.1¹⁾.



Key

- α angle of incident light
- λ wavelength of the radiation used
- n refractive index of crystal (n_1) and test specimen (n_2)
- 1 ATR-crystal
- 2 rubber material (test specimen)

Figure H.1 — Test specimen at the ATR-crystal and pathway of IR-beam

1) For more information refer to relevant literature or ISO surface infrared measurements. **Annex A1**

A1 H.2 Apparatus

H.2.1 Fourier transform infrared spectrometer (FTIR-spectrometer).

H.2.2 ATR-device.

H.3 Preparation of samples

H.3.1 For sample preparation, provide flat contact surfaces.

H.3.2 Cut the elastomeric part along an appropriate axis and take the cut edge as surface for measuring.

NOTE This provides an elastomeric spectrum without the influence of coatings. Also bands from silicone oil that is often applied to the surface of elastomeric closures, in this way will not disturb the spectrum of the elastomeric material.

H.4 Procedure

H.4.1 General

Conventional ATR is only appropriate for elastomeric parts, which have extended flat surfaces to be in contact with the ATR crystal.

Single bounce ATR is necessary if only small surfaces are available to be in contact with the ATR-crystal. Low contact areas give better results for strongly absorbing rubber samples (e.g. high filler content, carbon black as filler).

ATR equipment for an IR microscope provides also access to very small flat contact areas.

The refractive index of the crystal material determines among others (e.g. angle of incident light, wavelength) the depth of the penetration of the IR-light into the substrate.

The crystal material is chosen to be dependent on the absorbance characteristics of the analyte. In the case of highly absorbing elastomeric materials (e.g. carbon black filled formulations) a lower depth of penetration and/or a small contact area provide appropriate spectra.

H.4.2 Examination of the samples by ATR

Put the flat surface(s) of the elastomeric part(s) on the ATR crystal. Apply a constant (reproducible) pressure to get close contact.

Measure a spectrum from 700 cm⁻¹ to 3 800 cm⁻¹.

H.5 Expression of results

H.5.1 The result is a spectrum in transmission mode (% transmission) obtained as surface IR from wave-number 700 cm⁻¹ to 3 800 cm⁻¹. The range of spectrum may be slightly shifted depending on the crystal-material chosen. It can be compared to a reference spectrum with respect to the characteristic wave numbers.

H.5.2 Report the experimental conditions such as resolution, number of scans and bench purging. **A1**

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