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BSI Standards Publication

Rubber — Identification of antidegradants by gas chromatography/mass spectrometry

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National foreword

This British Standard is the UK implementation of ISO 10638:2017. It supersedes BS ISO 10638:2010, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/22, Testing and analysis of rubber.

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

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Rubber — Identification of antidegradants by gas chromatography/mass spectrometry

Caoutchouc — Identification des antidégradants par chromatographie en phase gazeuse/spectrométrie de masse

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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 documents 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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see the following URL: www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 10638:2010), which has been technically revised with the following changes:

— trap coolant has been removed from the reagents;

— a calibration procedure for the apparatus has been added;

— test conditions have been added;

— a data analysis clause (Clause 7) has been added.

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Introduction

Most rubber products contain antidegradants to extend the life of the product, the type of antidegradant depending on the service conditions to which a particular product will be exposed. Doubts are increasingly being expressed about the negative impact which rubber containing certain antidegradants can have on the environment. However, demonstrating the presence of antidegradants in rubber products is not easy.

There are methods of qualitative analysis, specified in International Standards such as ISO 4645, which use thin-layer chromatography. This requires a highly skilled operator with a great amount of knowledge and experience, as well as the use of standard reference materials.

The gas chromatography/mass spectrometry technique specified in this document is an efficient method suitable for identifying antidegradants contained in rubber products, as well as in the raw-rubber and the unvulcanized-rubber compounds used to make such products.

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Rubber — Identification of antidegradants by gas chromatography/mass spectrometry

WARNING 1 — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

WARNING 2 — Certain procedures specified in this document might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This document specifies a method using gas chromatography/mass spectrometry, for the identification of antidegradants in raw rubbers, latices, unvulcanized-rubber compounds and vulcanized-rubber products. It is applicable to the 31 types of antidegradant listed in Annex A. The method specified is qualitative and is not intended for quantitative analysis.

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 123, *Rubber latex — Sampling*

ISO 124, *Latex, rubber — Determination of total solids content*

ISO 1407:2011, *Rubber — Determination of solvent extract*

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

3 Terms and definitions

No terms and definitions are listed in this document.

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

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

Antidegradants are recovered from samples by thermal desorption or solvent extraction. For thermal-desorption method, the fume generated from heating test samples is to be analysed after being separated through the gas chromatograph/mass spectrometer which is connected to a heating and desorbing device. For solvent-extraction method, the extract of test samples obtained by solvent extraction process is to be separated and analysed by the gas chromatograph/mass spectrometer. The type of antidegradant recovered is identified by the mass spectrum that is produced after passing the antidegradant through a gas chromatograph and mass spectrometer connected in tandem. The retention index can be used as a supplementary means of identification if necessary.

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5 Thermal-desorption method

5.1 Reagents and materials

5.1.1 Gas chromatograph carrier gas, helium.

5.2 Apparatus

5.2.1 Thermal-desorption apparatus or equivalent, connectable to the gas chromatograph and which can be heated up to 350 °C.

NOTE An example of an equivalent apparatus is a pyrolyzer in which the heating temperature is kept low.

5.2.2 Gas chromatograph/mass spectrometer.

5.2.2.1 Gas chromatograph, as specified below:

— carrier gas flow rate: 1,0 ml/min to 2,0 ml/min;

— injector temperature: 300 °C to 350 °C;

— maximum oven temperature: 350 °C.

5.2.2.2 Column, as specified below:

— length: 25 m to 60 m;

— diameter: 0,25 mm to 0,35 mm;

— liquid phase: 5 % diphenyl-, 95 % polydimethylsiloxane;

— film thickness: 0,20 µm to 0,35 µm.

Other types of column (e.g. 100 % polydimethylsiloxane) may be used if the retention index given in Annex A, or determined as described in Annex C, is not utilized in the analysis.

5.2.2.3 Mass spectrometer, quadrupole mass spectrometer, magnetic-sector-type mass spectrometer or any other suitable type, having the characteristics specified below:

— interface temperature: 300 °C;

— ionization method: electron ionization;

— ion source temperature: 230 °C to 300 °C;

— ionizing voltage: 70 eV;

— scan range: mass/charge ratio (m/z): 50 to 600.

5.3 Sampling

5.3.1 In the case of latex, carry out sampling in accordance with ISO 123 and dry the sample in accordance with ISO 124.

5.3.2 In the case of raw rubber, carry out sampling in accordance with ISO 1795.

5.3.3 In the case of rubber compound which includes vulcanized or unvulcanized rubber, take out a sample that represents the whole (e.g. by sampling from the core part). Clean the surface of the sample

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5.4 Procedure

WARNING — Persons following the procedure specified in this subclause are expected to be familiar with analysis using gas chromatography/mass spectrometry. In addition, it is assumed that the gas chromatograph/mass spectrometer is operated in accordance with the manufacturer's instruction manual and that it is maintained in an optimum condition. Detailed procedures for operation of the equipment are therefore not included.

5.4.1 Adjust the mass/charge ratio (m/z) with calibration reference material in accordance with the instruction manual of the apparatus.

5.4.2 Set each apparatus as follows.

5.4.2.1 Thermal-desorption apparatus

— thermal-desorption temperature: 350 °C.

5.4.2.2 Gas chromatograph

— carrier gas flow rate: 1 ml/min to 2 ml/min;

— injector temperature: 300 °C to 350 °C;

— temperature programme:

a) initial temperature: 40 °C to 80 °C;

b) rate of temperature rise: 10 °C/min to 25 °C/min;

c) final temperature: 320 °C to 350 °C — set the final temperature of column oven at or below the highest temperature of column used;

d) retention time: 10 min to 30 min.

5.4.2.3 Mass spectrometer

— interface temperature: 300 °C;

— ionization method: electron ionization;

— ion source temperature: 230 °C to 300 °C;

— ionizing voltage: 70 eV;

— scan range: mass/charge ratio (m/z): 50 to 600.

5.4.3 Put approximately 0,2 mg to 2 mg of sample into a sample holder.

5.4.4 Put the sample holder (5.4.3) in the thermal-desorption apparatus and start the gas chromatography/mass spectrometry measurement in order to obtain the gas chromatogram and mass spectrum. For a more accurate identification, the gas-chromatographic retention index can be determined as described in Annex C.

6 Solvent-extraction method

6.1 Reagents and materials

6.1.1 **Extraction solvent**: acetone of analytical reagent grade.

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6.1.2 **Gas chromatograph carrier gas**, helium.

6.2 Apparatus

6.2.1 **Soxhlet extractor**, as specified in ISO 1407.

6.2.2 **Gas chromatograph/mass spectrometer**, as specified in 5.2.2.

6.3 Sampling

See 5.3.

6.4 Procedure

6.4.1 Adjust the mass/charge ratio (m/z) according to 5.4.1 and set gas chromatograph/mass spectrometer conditions as specified in 5.4.2.2 and 5.4.2.3.

6.4.2 Carry out a Soxhlet extraction, using acetone as solvent, on approximately 2 g of sample cut into cubes measuring 2 mm or less, continuing the extraction for approximately 8 h in accordance with ISO 1407:2011, method A.

If the 2 g sample does not give enough antidegradant, continue the extraction with more sample.

6.4.3 Concentrate the extract to between 10 ml and 20 ml and inject 1 µl of the concentrated extract into the gas chromatograph, and start the gas chromatography/mass spectrometry measurement in order to obtain the gas chromatogram and mass spectrum. For a more accurate identification, the gas-chromatographic retention index can be determined as described in Annex C.

If the raw-rubber or unvulcanized-rubber compound is soluble in acetone, the thermal-desorption method should be used instead of the solvent-extraction method.

7 Analysis

7.1 General

Analyse the data as specified in 7.2 for the detected substances. The characteristic mass/charge ratio (m/z) of each substance is provided in Annex A. The characteristic peak assignment and the mass spectrum are provided in Annex B.

7.2 Procedure

7.2.1 Display the mass spectrum of each chromatographic peak obtained by the procedure in 5.4 or 6.4.

7.2.2 Compare the obtained mass spectrum to Figures B.1 to B.31 to find the most similar pattern.

NOTE Mass-spectrum search systems are available on the market to help with this procedure.

7.2.3 Identify the antidegradants using Table A.1.

When gas-chromatographic retention indices have been obtained in the earlier steps, verify that they conform to the identified antidegradants' retention indices in Table A.1.

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7.3 Important observations for analysis

7.3.1 Make sure to analyse the whole spectrum including the major mass/charge ratio (m/z) and the relative intensity. The identification is more accurate when mass spectra and gas-chromatographic retention indices are examined together.

7.3.2 Note that more than one antidegradants are often found upon identification.

7.3.3 Some types of antidegradants are made of a mixture of several substances. In such cases, the gas chromatogram includes more than one set of peaks. Since the composition differs depending on the manufacturer or on the grade, the ratio of peak areas or heights will also differ.

7.3.4 There are also cases where only decomposition products of an antidegradant are detected. In such cases, it is necessary to identify the antidegradant by confirming the presence of a mass spectrum specific to each of the decomposition products.

8 Test report

The test report shall include the following information:

a) a full description of the sample and its origin;

b) the test method:

   1) a full reference to the test method used, i.e. the number of this document (ISO 10638);

   2) the procedure used to extract the antidegradant (thermal desorption or solvent extraction);

c) test details:

   1) the number of test samples analysed;

   2) details of any procedures not specified in this document;

d) the test result (i.e. the identity of the antidegradant found);

e) the date of the test.

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Annex A
(normative)

Antidegradants covered by this document

Table A.1 summarizes the name, the abbreviated terms, the CAS registry number, the characteristic components as detected by gas chromatography, the mass/charge ratio of the main peaks in the mass spectrum of each component, and the retention index of the characteristic components for the antidegradants covered by this document.

Table A.1 — Details of antidegradants covered by this document

Number[a]	Name	Abbreviated term (see ISO 6472)	CAS RN	Peak number[a]	Component detected	Characteristic mass/charge ratio(s) *m/z*		Retention index
B1	polymerized 2,2,4-trimethyl-1,2-dihydroquinoline	TMQ	26780–96–1	B1–1	2,2,4-trimethyl-1,2-dihydroquinoline	158	173	1 470
				B1–2	dimer	158	331	2 730
				B1–3	trimer	174	519	3 820
B2	6-ethoxy-1,2-dihydro-2,2,4-trimethylquinoline	ETMQ	91–53–2	B2–1	6-ethoxy-1,2-dihydro-2,2,4-trimethylquinoline	202	217	1 760
B3	acetone-diphenylamine condensate	ADPA	68412–48–6	B3–1	diphenylamine	169		1 650
				B3–2	isopropyl diphenylamine	196	211	1 910
				B3–3	9,9′-dimethylacridane	194	209	1 960
				B3–4	unknown	236	251	2 180
B4	reaction product of diphenylamine, aniline and acetone	—	—	B4–1	diphenylamine	169		1 640
				B4–2	9,9′-dimethylacridane	194	209	1 960
				B4–3	unknown	331	348	2 720
				B4–4	unknown	363	378	3 740
B5	*N*-phenyl-α-naphthylamine	PAN	90–30–2	B5–1	phenyl-1-naphthylamine	219		2 210
B6	alkylated diphenylamine	—	68921–45–9	B6–1	diphenylamine	169		1 650
				B6–2	4,4′-dibutyl-diphenylamine	210	281	2 330
				B6–3	4,4′-dioctyl-diphenylamine	322	393	3 000
B7	octylated diphenylamine	ODPA	106–67–7	B7–1	4,4′-dioctyl-diphenylamine	322	393	3 010

[a] See Annex B.

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Table A.1 *(continued)*

Number[a]	Name	Abbreviated term (see ISO 6472)	CAS RN	Peak number[a]	Component detected	Characteristic mass/charge ratio(s) *m/z*		Retention index
B8	4,4′-bis(α,α-dimethylbenzyl)-diphenylamine	—	10081–67–1	B8–1	4,4′-bis (*α,α*-dimethylbenzyl)-diphenylamine	390	405	3 660
B9	p-(p-toluenesulfonylamido)-diphenylamine	—	100–93–6	B9–1	*N*-phenyl-*p*-phenylenediamine	184		1 990
				B9–2	*p*-(*p*-toluenesulfonylamido)-diphenylamine	183	338	3 340
B10	N,N′-di-2-naphthyl-p-phenylenediamine	DNPD	93–46–9	B10–1	*N,N′*-di-2-naphthyl-*p*-phenylenediamine	360		4 120
B11	N,N′-diphenyl-p-phenylenediamine	DPPD	74–31–7	B11–1	*N,N′*-diphenyl-*p*-phenylenediamine	260		2 740
B12	N-isopropyl-N′-phenyl-p-phenylenediamine	IPPD	101–72–4	B12–1	*N*-phenyl-*N′*-isopropyl-*p*-phenylenediamine	211	226	2 150
B13	N-1,3-dimethylbutyl-N′-phenyl-p-phenylenediamine	6PPD	793–24–8	B13–1	*N*-phenyl-*N′*-(1,3-dimethylbutyl)-*p*-phenylenediamine	211	268	2 390
B14	N-(1-methylheptyl)-N′-phenyl-p-phenylenediamine	—	15233–47–3	B14–1	*N*-(1-methylheptyl)-*N′*-phenyl-*p*-phenylenediamine	211	296	2 660
B15	mixed diaryl-p-phenylenediamine	—	68953–84–4	B15–1	*N,N′*-diphenyl-*p*-phenylenediamine	260		2 730
				B15–2	*N,N′*-dimethylphenyl-*p*-phenylenediamine	288		2 810
				B15–3	*N,N′*-dimethylphenyl-*p*-phenylenediamine (isomer)	288		2 880
B16	2,6-di-tert-butyl-4-methylphenol	BHT	128–37–0	B16–1	2,6-di-*tert*-butyl-4-methylphenol	205	220	1 530
B17	2,6-di-tert-butyl-4-ethylphenol	—	4130–42–1	B17–1	2,6-di-*tert*-butyl-4-ethylphenol	219	234	1 580

[a] See Annex B.

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Table A.1 *(continued)*

Number[a]	Name	Abbreviated term (see ISO 6472)	CAS RN	Peak number[a]	Component detected	Characteristic mass/charge ratio(s) *m/z*		Retention index
B18	styrenated phenol	SPH	61788–44–1	B18–1	*α*-methylbenzylphenol	183	198	1 740
				B18–2	di-*α*-methylbenzylphenol	287	302	2 530
				B18–3	tri-*α*-methylbenzylphenol	391	406	3 080
B19	n-octadecyl-3-(4-hydroxy-3′,5′-di-tert-butylphenyl) propionate	—	2082–79–3	B19–1	*n*-octadecyl-3-(4-hydroxy-3′,5′-di-*t*-butylphenyl) propionate	515	530	3 630
B20	2-t-butyl-6-(3-t-butyl-2-hydroxy-5-methylbenzyl)-4-methylphenyl acrylate	—	61167–58–6	B20–1	2-*t*-butyl-6-(3-*t*-butyl-2-hydroxy-5-methylbenzyl)-4-methylphenyl acrylate	361	394	2 610
B21	2,2′-methylene-bis(4-ethyl-6-tert-butylphenol)	*o*-MBp24	88–24–4	B21–1	2,2′-methylene-bis(4-ethyl-6-*tert*-butylphenol)	191	368	2 550
B22	2,2′-methylene-bis(4-methyl-6-tert-butylphenol)	*o*-MBp14	119–47–1	B22–1	2,2′-methylene-bis(4-methyl-6-*tert*-butylphenol)	177	340	2 460
B23	4,4′-butylidene-bis(6-tert-butyl-m-cresol)	*p*-BBp14	85–60–9	B23–1	4,4′-butylidene bis(3-methyl-6-*tert*-butylphenol)	339	382	2 740
B24	4,4′-thio-bis(2-tert-butyl-m-cresol)	*p*-TBp14	96–69–5	B24–1	4,4′-thio-bis(3-methyl-6-*tert*-butylphenol)	343	358	2 820
B25	butylated reaction product of p-cresol and dicydopentadiene	—	68610–51–5	B25–1	2,2′-cyclopentadiene-bis(4-methyl-6-*tert*-butyl-phenol)	445	460	3 750

[a] See Annex B.

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Table A.1 *(continued)*

Number[a]	Name	Abbreviated term (see ISO 6472)	CAS RN	Peak number[a]	Component detected	Characteristic mass/charge ratio(s) *m/z*		Retention index
B26	2,2′-methylene-bis[6-(1-methylcyclohexyl)-p-cresol]	*o*-MBp1(1C)	77–62–3	B26–1	2,2′-methylene-bis[6-(1-methylcyclohexyl-*p*-cresol)]	217	420	3 330
B27	2,5-di-tert-butyl-hydroquinone	DBHQ	88–58–4	B27–1	unknown	205	220	1 480
				B27–2	2,5-di-*tert*-butyl-hydroquinone	207	222	1 810
B28	2,5-di-tert-amyl-hydroquinone	DAHQ	79–74–3	B28–1	2,5-di-pentyl-*p*-benzoquinone	177	248	1 680
				B28–2	2,5-di-*tert*-amyl-hydroquinone	221	250	1 980
B29	tributyl thiourea	—	2422–88–0	B29–1	di-*n*-butylamine	86	129	960
				B29–2	butylisothiocyanate	72	115	1 000
B30	dilauryl thiodipropionate	DLTDP	123–28–4	B30–1	dilauryl thiodipropionate	329	514	3 630
B31	butyl hydroxyanisole	BHA	25013–16–5	B31–1	3-*tert*-butyl-4-hydroxyanisole	165	180	1 500

[a] See Annex B.

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Annex B
(informative)

Chromatograms and mass spectra

Chromatograms and mass spectra of antidegradants covered by this document are shown in Figures B.1 to B.31. The chromatograms and mass spectra were obtained under the following conditions:

a) Thermal-desorption apparatus: Double-Shot Pyrolyzer Model PY-2020iD[1]:

— thermal-desorption temperature: 350 °C.

b) Gas chromatograph/mass spectrometer: Agilent 6890[2] and Agilent 5973[2]:

— carrier gas flow rate: 1,0 ml/min;

— total flow rate: 300 ml/min (split ratio 1/300);

— injector temperature: 320 °C;

— oven temperature programme: initial temperature 40 °C, heating by 20 °C/min up to 320 °C, maintained at 320 °C for 10 min;

— interface temperature: 300 °C;

— ionization method: electron ionization;

— ion source temperature: 250 °C;

— ionizing voltage: 70 eV;

— scan range: mass/charge ratio (m/z) 29 to 600.

c) Column: Ultra ALLOY-5 (Frontier-LAB)[3]:

— length: 30 m;

— diameter: 0,25 mm;

— liquid phase: 5 % diphenyl-, 95 % polydimethylsiloxane;

— film thickness: 0,25 µm.

1) Double-Shot Pyrolyzer Model PY-2020iD is a model of thermal-desorption apparatus supplied by Frontier Lab. 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.

2) Agilent 6890 and Agilent 5973 are models of gas chromatograph/mass spectrometer supplied by Agilent Technologies. 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.

3) Ultra ALLOY-5 is a registered trademark for stainless steel columns supplied by Frontier Lab. 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.

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