INTERNATIONAL STANDARD

First edition 2010-07-01

Rubber — Identification of antidegradants by gas chromatography/mass spectrometry

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

iTeh STANDARD PREVIEW (standards.iteh.ai)

<u>ISO 10638:2010</u> https://standards.iteh.ai/catalog/standards/sist/5f79dfe0-166c-4e68-b974-39ef6d1a8260/iso-10638-2010



Reference number ISO 10638:2010(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW (standards.iteh.ai)

<u>ISO 10638:2010</u> https://standards.iteh.ai/catalog/standards/sist/5f79dfe0-166c-4e68-b974-39ef6d1a8260/iso-10638-2010



COPYRIGHT PROTECTED DOCUMENT

© ISO 2010

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

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

Contents

Page

Forew	vord	iv
Introd	duction	v
1	Scope	1
2	Normative references	1
3	Principle	1
4	Thermal-desorption method	2
4.1	Reagents and materials	
4.2	Apparatus	2
4.3	Sampling	3
4.4	Procedure	3
5	Solvent-extraction method	3
5.1	Reagents and materials	
5.2	Apparatus	3
5.3	Sampling	
5.4	Procedure	3
6	Procedure Determination of gas-chromatographic retention index	4
7	Analysis (standards.iteh.ai)	4
8	Test report ISO 10638:2010	4
Annex	x A (normative), Antidegradants covered by this International Standard	5
Annex	x B (informative) Chromatograms and mass spectra 010	8
Annex	x C (normative) Determination of gas-chromatographic retention index	40
Biblio	ography	43

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 10638 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

iTeh STANDARD PREVIEW (standards.iteh.ai)

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 International Standard 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.

iTeh STANDARD PREVIEW (standards.iteh.ai)

iTeh STANDARD PREVIEW (standards.iteh.ai)

Rubber — Identification of antidegradants by gas chromatography/mass spectrometry

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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.

CAUTION — Certain procedures specified in this International Standard may 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 International Standard specifies a qualitative 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. Users should note that the method specified is a qualitative one and is not intended for quantitative analysis.

NOTE Persons using this International Standard are expected to be familiar with procedures of 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 10

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 123, Rubber latex Sampling
- ISO 124, Latex, rubber Determination of total solids content
- ISO 1407, Rubber Determination of solvent extract
- ISO 1795, Rubber, raw natural and raw synthetic Sampling and further preparative procedures
- ISO 4661-2, Rubber, vulcanized Preparation of samples and test pieces Part 2: Chemical tests

3 Principle

Antidegradants are recovered from samples by thermal desorption or solvent extraction. 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.

4 Thermal-desorption method

4.1 Reagents and materials

4.1.1 Trap coolant, e.g. liquid nitrogen, used only when required by the particular thermal-desorption apparatus in use.

4.1.2 Gas chromatograph carrier gas: helium.

4.2 Apparatus

4.2.1 Thermal-desorption apparatus or equivalent, connectable to the gas chromatograph and which can be heated up to 340 °C.

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

4.2.2 Trap apparatus, used only when required by the particular thermal-desorption apparatus in use.

4.2.3 Gas chromatograph/mass spectrometer.

4.2.3.1 Gas chromatograph, as specified below:

- Column:
 - length: 25 m to 60 m; **iTeh STANDARD PREVIEW**
 - diameter: 0,25 mm to 0,35 mm; (standards.iteh.ai)
 - liquid phase: 5 % diphenyl-, 95 % polydimethylsiloxane; 0
 - https://standards.iteh.ai/catalog/standards/sist/5f79dfe0-166c-4e68-b974-
 - film thickness: 0,20 μm to 0,35 μm. <u>39ef6d1a8260/iso-10638-2010</u>

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.

- Carrier gas flow rate: 1,0 ml/min to 2,0 ml/min.
- Injector temperature: 300 °C to 350 °C.
- Oven temperature programme: initial temperature 40 °C, heating at 20 °C/min up to 320 °C, maintained at 320 °C for 10 min.

4.2.3.2 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.

4.3 Sampling

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

4.3.2 In the case of raw rubber and rubber compounds, carry out sampling in accordance with ISO 1795 or ISO 4661-2, respectively.

4.4 Procedure

4.4.1 Put approximately 1 mg to 2 mg of sample into a sample holder (cup or tube).

4.4.2 Put this test sample in the thermal-desorption apparatus, maintained at 340 °C, and start the gas chromatography/mass spectrometry measurement in order to obtain the gas chromatogram and mass spectrum.

If a trap apparatus is used, heat the sample at 340 °C for 1 min and trap the volatile components generated during the heating period in the trap cooled by liquid nitrogen. On completion of the heating period, stop supplying the liquid nitrogen and start the gas chromatography/mass spectrometry measurement in order to obtain the gas chromatogram and mass spectrum.

5 Solvent-extraction method

5.1 Reagents and materials TANDARD PREVIEW

- 5.1.1 Extraction solvent: acetone of analytical reagent gradel)
- 5.1.2 Gas chromatograph carrier gas: helium_{0638:2010}

https://standards.iteh.ai/catalog/standards/sist/5f79dfe0-166c-4e68-b974-

- **5.2** Apparatus 39ef6d1a8260/iso-10638-2010
- 5.2.1 Soxhlet extractor, as specified in ISO 1407.
- 5.2.2 Gas chromatograph/mass spectrometer, as specified in 4.2.3.

5.3 Sampling

See 4.3.

5.4 Procedure

5.4.1 Carry out a Soxhlet extraction, using acetone as solvent, on approximately 2 g of sample that has been cut into cubes measuring 2 mm or less, continuing the extraction for approximately 8 h.

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

5.4.2 Concentrate the extract to between 10 ml and 20 ml and inject 1 μ l of the concentrated extract into the gas chromatograph/mass spectrometer.

NOTE The amount of concentrated extract injected depends on the amount of antidegradant in the rubber.

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.

6 Determination of gas-chromatographic retention index

See Annex C.

7 Analysis

Gas chromatograms and mass spectra of the antidegradants covered by this International Standard are shown in Figures B.1 to B.31, in which the gas-chromatographic peaks characteristic of each antidegradant are indicated. Further details of these peaks are given in Table A.1, including the identity of each component concerned, the retention index of the component and the mass/charge ratios (m/z) of the main peaks in the mass spectrum of each component.

For the particular antidegradant being analysed, display the mass spectrum corresponding to each of the characteristic peaks in the chromatogram and verify that these mass spectra are identical to the mass spectra shown in Annex B.

Note that the retention indices are given for guidance only. Although they are useful for predicting retention times, the identity of an antidegradant shall be confirmed primarily by checking the mass spectra obtained.

Some types of antidegradant are a mixture of several chemical compounds rather than a single compound. In such cases, the gas chromatogram will include more than one set of peaks. The ratios of the quantities of each compound present (which will affect the peak ratios) will depend on the manufacturer and the grade.

There are also cases in which 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 of the antidegradant. near ds.iteh.ai)

Another case which will need to be taken into consideration is that in which more than one antidegradant has been included in the rubber compound. <u>ISO 10638:2010</u>

https://standards.iteh.ai/catalog/standards/sist/5f79dfe0-166c-4e68-b974-39ef6d1a8260/iso-10638-2010

8 Test report

The test report shall include the following particulars:

- a) a full description of the sample and its origin;
- b) test method:
 - 1) a full reference to the test method used, i.e. the number of this International Standard;
 - 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 International Standard;
- d) the test result (i.e. the identity of the antidegradant found);
- e) the date of the test.

Annex A

(normative)

Antidegradants covered by this International Standard

The name, the abbreviation, the CAS registry number, the characteristic components as detected by gas chromatography, the mass/charge ratios of the main peaks in the mass spectrum of each of these components, and the retention indices of the characteristic components are summarized, for the antidegradants covered by this International Standard, in Table A.1.

Number ^a	Name	Abbreviation (see ISO 6472)	CAS RN	Peak number ^a	Component detected	Characteristic mass/charge ratio(s) m/z		Retention index
	polymerized 2,2,4-trimethyl-			B1-1	2,2,4-trimethyl- 1,2-dihydroquinoline	158	173	1 466
B1	1,2-dihydroquinoline	TMQ	26780-96-1	B1-2	dimer	158	331	2 725
				B1-3	trimer	174	519	3 823
B2	6-ethoxy-1,2-dihydro-2,2,4- trimethylquinoline	SETMON	91-53-2) B2- R	6-ethoxy-1,2-dihydro- 2,2,4-trimethylquinoline	202	217	1 762
		(stand	lards.	ards. 1831. appenylamine 169		<u> </u>	1 645	
B3	acetone-diphenylamine	ADPA T	568412-48-6	B3-2	isopropyl diphenylamine	196	211	1 911
63	condensate	ds.iteh.ai/catalo)10 B3-3	9,9'-dimethylacridane	194	209	1 963
	nups//standal		a8260/iso-10	63 <mark>8324</mark> 010	unknown	236	251	2 175
				B4-1	diphenylamine	16	69	1 644
B4	reaction product of diphenylamine, aniline and acetone	_	_	B4-2	9,9'-dimethylacridane	194	209	1 963
D4				B4-3	unknown	331	348	2 722
				B4-4	unknown	363	378	3 738
B5	<i>N</i> -phenyl- <i>a</i> -naphthylamine	PAN	90-30-2	B5-1	phenyl-1-naphthylamine	219		2 205
	alkylated diphenylamine	_	68921-45-9	B6-1	diphenylamine	16	69	1 645
B6				B6-2	4,4'-dibutyl-diphenylamine	210	281	2 326
				B6-3	4,4'-dioctyl-diphenylamine	322	393	3 004
B7	octylated diphenylamine	ODPA	106-67-7	B7-1	4,4'-dioctyl-diphenylamine	322	393	3 009
B8	4,4′-bis(<i>α</i> , <i>α</i> -dimethylbenzyl)- diphenylamine	_	10081-67-1	B8-1	4,4'-bis (α , α -dimethylbenzyl)- diphenylamine	390	405	3 655
В9	<i>p</i> -(<i>p</i> -toluenesulfonylamido)- diphenylamine	_	100-93-6	B9-1	<i>N</i> -phenyl- <i>p</i> -phenylenediamine	184		1 986
БЯ				B9-2	<i>p</i> -(<i>p</i> -toluenesulfonylamido)- diphenylamine	183	338	3 337
B10	<i>N,N</i> '-di-2-naphthyl- <i>p</i> -phenylenediamine	DNPD	93-46-9	B10-1	<i>N,N</i> '-di-2-naphthyl- <i>p</i> -phenylenediamine	360		4 124
B11	<i>N,N</i> '-diphenyl- <i>p</i> -phenylenediamine	DPPD	74-31-7	B11-1	<i>N,N</i> ′-diphenyl- <i>p</i> -phenylenediamine	260		2 736

Table A.1 — Details of antidegradants covered by this International Standard

$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	Number ^a	Name	Abbreviation (see ISO 6472)	CAS RN	Peak number ^a	Component detected	Characteristic mass/charge ratio(s) m/z		Retention index
B13 <i>μ</i> -phenylenediamine 6PPD 793-24-8 B13.1 <i>μ</i> -(1,3-dimetrylbutyl)- <i>μ</i> -phenylenediamine 211 288 2 391 B14 <i>μ</i> -(1-metrylbeptyl)- <i>μ</i> -phenylenediamine - 15233-47.3 B14.1 <i>μ</i> -(1-metrylbeptyl)- <i>μ</i> -phenylenediamine 211 288 2 657 B15 <i>μ</i> -phenylenediamine - 15233-47.3 B14.1 <i>μ</i> -dimetrylbeptyl- <i>μ</i> -phenylenediamine 211 298 2 657 B15 <i>μ</i> -phenylenediamine - 68953-844 B15.1 <i>M</i> -dimetrylbenyl- <i>μ</i> -phenylenediamine 288 2 880 B16 2.6-di-fert-butyl- 4-metrylphenol BHT 128-37-0 B161-1 2.6-di-fert-butyl- 4-metrylphenol 205 220 1528 B17 2.6-di-fert-butyl- 4-metrylphenol B17.1 2.6-di-fert-butyl- 4-metrylphenol 213 108 174 B18 styrenated phenol SPH 6187.4 B18.1 center/butyl- 4-metrylphen	B12		IPPD	101-72-4	B12-1		211	226	2 153
B14 Nr-phenyl- p-phenylenediamine 15233-47-3 p-phenylenediamine B14-1 B14-1 Nr-phenyl- p-phenylenediamine 211 296 2 657 B15 mixed diaryl- p-phenylenediamine 68953-84-4 B15-2 N/N-diphenyl- p-phenylenediamine 260 2 728 B16 2.6-di-fert-butyl- +-methylphenol BHT 128-37-0 B16-1 R-fert-butyl- p-phenylenediamine 2.8 2 880 B17 2.6-di-fert-butyl- +-methylphenol BHT 128-37-0 B16-1 2.6-di-fert-butyl- di-methylphenol 205 220 1 528 B18 styrenated phenol SPH SPH 819-1 2.6-di-fert-butyl- di-methylphenylone 130 198 1744 B18 styrenated phenol SPH SPH SPH 819-1 di-methylphenylone 391 406 3082 B19 2.7-di-tert-butyl- methylphenyl acrylate 61167-58-6 B20-1 2-butyl-6(-3-butyl-2- methylene-bis(4- methyl-6(-3-butyl-2- methylene-bis(4- methyl-6(-3-butyl-2- methylene-bis(4- methylene) 361 394 2 608 B22 2.2-methylene-bis(4- m	B13	N'-phenyl-	6PPD	793-24-8	B13-1	N'-(1,3-dimethylbutyl)-	211	268	2 391
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	B14	N'-phenyl-	_	15233-47-3	B14-1	N'-phenyl-	211	296	2 657
B15 Initial display- printing display- display- B16 Image: mail of the state printing display- printing display- printing display- printing display- printing display- printing display- printing display- printing display- printing display- display- B17 Image: mail display- printing display- biling display- biling display- printing display- biling display- biling display- biling display- biling display- biling display- biling display- biling display- B18 Image: mail display- biling display- bilin					B15-1				2 728
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	B15		_	68953-84-4	B15-2				2 810
B10 4-methylphenol 205 220 1 528 B17 2.6-di-tert-butyl- 4-ethylphenol 2.6-di-tert-butyl- 4-ethylphenol 2.6-di-tert-butyl- 4-ethylphenol 2.19 2.34 1 577 B18 styrenated phenol Ten ST 4130-42-1 500 B17-1 500 Cl-tert-butyl- 4-ethylphenol 2.6-di-tert-butyl- 4-ethylphenol 2.19 2.34 1 577 B18 styrenated phenol SPH 61788-44 B18-1 armethylphenol 183 198 1 744 B19 3',5'-di-tert-butylphenol 2.87 0.02 2.530 B19 3',5'-di-tert-butylphenol 2.87 0.02 2.550 B20 n-createlystant armethylopazylphenol 2.87 0.02 2.45-butyl-6(-3-4-butyl-2- hydroxy-5-methylbenzyl)-4 0.06 3.082 2.5-di-tert-butylphenol 3.61 3.94 2.608 B21 2.2'-tmethylene-bis(4- methyl-6-fert-butylphenol) o-MBp24 88-24-4 B21-1 2.2'-tmethylene-bis(4- methyl-6-fert-butylphenol) 3.68 2.608 B22 2.2'-tmethylene-bis(4- methyl-6-fert-butylphenol) o-MBp24 </td <td></td> <td>p pronjonou unino</td> <td></td> <td></td> <td>B15-3</td> <td>p-phenylenediamine</td> <td colspan="2">288</td> <td>2 880</td>		p pronjonou unino			B15-3	p-phenylenediamine	288		2 880
B17 4-ethylphenol Teh ST 30:4-1 B18 4-ethylphenol 219 2.9 1.07 B18 styrenated phenol SPH 61788.444 B18-1 a-methylbenzylphenol 183 198 1.744 B18 styrenated phenol SPH 61788.444 B18-2 dra-methylbenzylphenol 287 302 2.530 B19 a'.5'-di-tert-butylphenyl)	B16		BHT	128-37-0	B16-1		205	220	1 528
B18 styrenated phenol SPH 61788.44.4 B18.2 dr.amethylbenzylphenol 287 302 2 530 B19 n-octadecyl-3-(4-hydroxymes/standards.ici 3/5'-di-lert-butylphenyl)	B17		Teh ST	4130-42-1	B17-1		219	234	1 577
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$					B18-1	<i>a</i> -methylbenzylphenol	183	198	1 744
B19 n-octadecyl-3-(4-hydroxyrpp 3',5'-di-tert-butylphenyl) propionate /standards.itel alcalandes Indirects of propertodecyl-3-(4-hydroxyrpp 3',5'-di-tert-butylphenyl) 515 530 3 628 B20 2-f-butyl-6-(3-f-butyl-2- hydroxy-5-methylbenzyl)-4- methylphenyl acrylate — 61167-58-6 B20-1 2-f-butyl-6-(3-f-butyl-2- hydroxy-5-methylbenzyl)-4- methylphenyl acrylate 361 394 2 608 B21 2.2'-methylene-bis(4- etrd-butylphenol) o-MBp24 88-24-4 B21-1 2.2'-methylene-bis(4- ethyl-6-ferf-butylphenol) 191 368 2 549 B22 2.2'-methylene-bis(4- methyl-6-ferf-butylphenol) o-MBp14 119-47-1 B22-1 2.2'-methylene-bis(4- ethyl-6-ferf-butylphenol) 177 340 2 457 B23 4.4'-butylidene-bis(6-ferf- butyl-m-cresol) p-BBp14 86-60-9 B23-1 4.4'-butylidene-bis(3- methyl-6-ferf-butylphenol) 339 382 2 736 B24 4.4'-thio-bis(2-ferf-butyl- m-cresol) p-TBp14 96-69-5 B24-1 4.4'-thio-bis(3-methyl- 6-ferf-butylphenol) 343 358 2 820 B25 butylated reaction product of p-cresol and dicydopentadiene — 68610-51-5	B18	styrenated phenol	SPH (S	61788-44-1	B18-2	di- <i>a</i> -methylbenzylphenol	287	302	2 530
$\begin{array}{c c c c c c c c c c c c c c c c c c c $				100	B18-3	tri- <i>a</i> -methylbenzylphenol	391	406	3 082
B20 hydroxy-5-methylbenzyl)-4- methylphenyl acrylate 61167-58-6 B20-1 hydroxy-5-methylbenzyl)-4- 4-methylphenyl acrylate 361 394 2 608 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 2 549 B22 2.2'-methylene-bis(4- ethyl-6-tert-butylphenol) o-MBp14 119-47-1 B22-1 2.2'-methylene-bis(4- ethyl-6-tert-butylphenol) 177 340 2 457 B23 4.4'-butylidene-bis(6-tert- butyl-m-cresol) ρ -BBp14 85-60-9 B23-1 4.4'-butylidene bis(3- methyl-6-tert-butylphenol) 339 382 2 736 B24 4.4'-thio-bis(2-tert-butyl- m-cresol) ρ -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 $$ 68610-51-5 B25-1 2.2'-cyclopentadiene- bis(6-(1-methylcyclohexyl- p-cresol]) 217 420 3 327 B26 2.2'-methylene- bis(6-(1-methylcyclohexyl- p-cresol] $0-MBp1(1C)$ 77-62-3 B26-1 2.2'-methylene- bis(6-(1-methylcyclohexyl- p-cresol]) 217 420	B19	3',5'-di-tert-butylphenyl)	://standards.itel	1.ai/catalog/st	andards/siz 60/1s0-100	3',5'-di-t-butylphenyl)	4- 515	530	3 628
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	B20	hydroxy-5-methylbenzyl)-4-	_	61167-58-6	B20-1	hydroxy-5-methylbenzyl)-	361	394	2 608
B22 methyl-6-tert-butylphenol) OWB p14 Part 1 B22-1 methyl-6-tert-butylphenol) Part 1 S40 2437 B23 4,4'-butylidene-bis(6-tert- butyl-m-cresol) p -BBp14 85-60-9 B23-1 4,4'-butylidene bis(3- methyl-6-tert-butylphenol) 339 382 2 736 B24 4,4'-thio-bis(2-tert-butyl- m-cresol) p -TBp14 96-69-5 B24-1 4,4'-thio-bis(3-methyl- 6-tert-butylphenol) 343 358 2 820 B25 butylated reaction product of p-cresol and dicydopentadiene $$ 68610-51-5 B25-1 $2,2'$ -cyclopentadiene- bis(4-methyl-6-tert-butyl- bphenol) 445 460 3 746 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 3 327 B27 $2,5$ -di-tert-butyl- hydroquinone $DBHQ$ $88-58-4$ B27-2 $2,5$ -di-tert-butyl- hydroquinone 207 222 1 483 B28 $2,5$ -di-tert-amyl- hydroquinone $DAHQ$ $79-74-3$ B28-1 $2,5$ -di-tert-amyl- p-benzoquinone 177 248 1 680 B28 $2,5$ -di-tert-amyl- hydroqu	B21		o-MBp24	88-24-4	B21-1		191	368	2 549
B23 butyl-m-cresol p -BBp14 85-60-9 B23-1 methyl-6-tert-butylphenol 339 382 2736 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 3 746 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 3 327 B27 2.5 -di-tert-butyl- hydroquinone $DBHQ$ $88-58-4$ B27-1 unknown 205 220 1 483 B28 2.5 -di-tert-amyl- hydroquinone $DAHQ$ $79-74-3$ B28-1 2.5 -di-tert-butyl- p-benzoquinone 207 222 1 840 B28 2.5 -di-tert-amyl- hydroquinone $DAHQ$ $79-74-3$ B28-1 2.5 -di-tert-amyl- p-benzoquinone 177 248 1 680	B22		o-MBp14	119-47-1	B22-1		177	340	2 457
$\frac{1}{P-1} \frac{1}{P-1} \frac{1}$	B23		<i>p</i> -BBp14	85-60-9	B23-1		339	382	2 736
B25 of p-cresol and dicydopentadiene — 68610-51-5 B25-1 bis(4-methyl-6-tert-butyl-phenol) 445 460 3 746 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 3 327 B27 2,5-di-tert-butyl-hydroquinone DBHQ 88-58-4 B27-1 unknown 205 220 1 483 B28 2,5-di-tert-amyl-hydroquinone DBHQ 79-74-3 B28-1 2,5-di-pentyl-p-benzoquinone 177 248 1 680 B28 2,5-di-tert-amyl-hydroquinone DAHQ 79-74-3 B28-1 2,5-di-tert-amyl-p-benzoquinone 177 248 1 680	B24		<i>p</i> -TBp14	96-69-5	B24-1		343	358	2 820
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	B25	of p-cresol and	_	68610-51-5	B25-1	bis(4-methyl-6-tert-butyl-	445	460	3 746
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	B26	bis[6-(1-methylcyclohexyl)-	o-MBp1(1C)	77-62-3	B26-1	bis[6-(1-methylcyclohexyl-	217	420	3 327
$\frac{1}{1} \frac{1}{1} \frac{1}$			DBHQ	88-58-4	B27-1	unknown	205	220	1 483
B28 2,5-di-tert-amyl- hydroquinone DAHQ 79-74-3 B28-1 p-benzoquinone 177 248 1 680 B28 DAHQ 79-74-3 B28-1 p-benzoquinone 177 248 1 680	B27				B27-2		207	222	1 814
nyaroquinone 231 250 1.022	B28		DAHQ	79-74-3	B28-1		177	248	1 680
	520				B28-2		221	250	1 982

Table A.1 (continued)

Number ^a	Name	Abbreviation (see ISO 6472)	CAS RN	Peak number ^a	Component detected	Characteristic mass/charge ratio(s) m/z		Retention index
B29	tributyl thiourea		2422-88-0	B29-1	di- <i>n</i> -butylamine	86	129	960
B29			2722-00-0	B29-2	butylisothiocyanate	72	115	1 003
B30	dilauryl thiodipropionate	DLTDP	123-28-4	B30-1	dilauryl thiodipropionate	329	514	3 629
B31	butyl hydroxyanisole	BHA	25013-16-5	B31-1	3- <i>tert</i> -butyl- 4-hydroxyanisole	165	180	1 495
^a See A	Annex B.	•						

Table A.1 (continued)

iTeh STANDARD PREVIEW (standards.iteh.ai)

Annex B

(informative)

Chromatograms and mass spectra

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

- a) Thermal-desorption apparatus: Frontier-LAB model PY-2020 iD:
 - thermal-desorption temperature: 350 °C.
- b) Trap apparatus: not used.
- c) Gas chromatograph/mass spectrometer: Agilent 6890 and Agilent 5973:
 - 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; (standards.iteh.ai)
 - interface temperature: 300 °C;

e: 300 C, <u>ISO 10638:2010</u> https://standards.iteh.ai/catalog/standards/sist/5f79dfe0-166c-4e68-b974-

- ionization method: electron ionization;9ef6d1a8260/iso-10638-2010
- ion source temperature: 250 °C;
- ionizing voltage: 70 eV;
- scan range: mass/charge ratio (m/z) 29 to 600.
- d) Column: Ultra ALLOY-5 (Frontier-LAB):
 - length: 30 m;
 - diameter: 0,25 mm;
 - liquid phase: 5 % diphenyl-, 95 % polydimethylsiloxane;
 - film thickness: 0,25 μm.



Figure B.1 — Polymerized 2,2,4-trimethyl-1,2-dihydroquinoline