

---

---

**Textiles — Determination of  
deodorant property —**

**Part 3:  
Gas chromatography method**

*Textiles — Détermination des propriétés de neutralisation d'odeurs —  
Partie 3: Méthode par chromatographie en phase gazeuse*





**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2014

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested 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](mailto:copyright@iso.org)  
Web [www.iso.org](http://www.iso.org)

Published in Switzerland

# Contents

	Page
<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Principle</b> .....	<b>1</b>
<b>4 Reagents</b> .....	<b>1</b>
<b>5 Materials and apparatus</b> .....	<b>2</b>
<b>6 Testing environment</b> .....	<b>2</b>
<b>7 Test procedure</b> .....	<b>2</b>
7.1 General .....	2
7.2 Preparation of specimen .....	2
7.3 Method A .....	2
7.4 Method B .....	4
7.5 Concentration measurement of testing gas by GC .....	5
<b>8 Calculation of odour reduction rate</b> .....	<b>5</b>
<b>Annex A (informative) GC parameter</b> .....	<b>6</b>
<b>Annex B (informative) Round-robin test result</b> .....	<b>7</b>

## 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](http://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](http://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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 38, *Textiles*.

ISO 17299 consists of the following parts, under the general title *Textiles — Determination of deodorant property*:

- *Part 1: General principle*
- *Part 2: Detector tube method*
- *Part 3: Gas chromatography method*
- *Part 4: Condensation sampling analysis*
- *Part 5: Metal-oxide semiconductor sensors method*

## Introduction

This part of ISO 17299 describes a gas chromatography testing method for concentration measurement of odour chemicals in deodorant testing. This is the most general testing method that could be carried out in many testing laboratories globally if they have gas chromatography.

To avoid duplication, the major stream of the testing procedure is described in ISO 17299-1. This part of ISO 17299 describes a specific procedure for the gas chromatography testing method.



# Textiles — Determination of deodorant property —

## Part 3: Gas chromatography method

### 1 Scope

This part of ISO 17299 specifies a gas chromatography test method for the deodorant testing of all textile products. This method applies to the odour component chemicals, such as indole, isovaleric acid, nonenal, and acetic acid with added sodium chloride (NaCl). Two preparation methods are described in this test method:

- an odour chemical is applied in a container avoiding contact with a specimen in method A. Each chemical is tested separately;
- an odour chemical is injected directly on to the specimen in a container in method B. The chemicals tested in this method are a mixture of acetic acid and sodium chloride (NaCl).

### 2 Normative references

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

ISO 139, *Textiles — Standard atmospheres for conditioning and testing*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

### 3 Principle

Concentration of the gaseous odour component chemicals of the gas in containers with or without a test specimen after a designated contacting time is measured by gas chromatography (GC). The reduction rate of concentration of odour chemicals in the container is calculated from the concentration data with a specimen and without a specimen.

### 4 Reagents

Unless otherwise specified, analytical grades shall be used.

- 4.1 **Acetic acid (CH<sub>3</sub>COOH)**, reagent with a purity of 99,7 %.
- 4.2 **Indole (C<sub>8</sub>H<sub>7</sub>N)**, reagent.
- 4.3 **Isovaleric acid**, reagent with a purity of 98,0 %.
- 4.4 **2-Nonenal (C<sub>9</sub>H<sub>16</sub>O)**, reagent with a purity of 95,0 %.
- 4.5 **Diluent gas**, nitrogen gas from a nitrogen gas cylinder with a purity of at least 99,99 %.
- 4.6 **NaCl**, reagent grade.
- 4.7 **Ethanol**, reagent grade.
- 4.8 **Water**, grade 3 of ISO 3696.

## 5 Materials and apparatus

- 5.1 **Conical flask**, with a capacity of 500 ml, made of glass.
- 5.2 **Sealing film**, capable of expanding with no air permeability [e.g. Parafilm<sup>1</sup>].
- 5.3 **Injection vial**, with a capacity of 22 ml, with a rubber stopper and aluminium cap.
- 5.4 **Injection syringe**, capable of injecting at 8 µl and 850 µl in volume.
- 5.5 **Gastight syringe**.
- 5.6 **Heating oven**, capable of heating at 80 °C ± 2 °C for 30 min.
- 5.7 **Gas chromatography (GC) apparatus**, with a flame ionization detector (FID) or mass selective detector (MSD).
- 5.8 **Rubber stopper and aluminium cap**.

## 6 Testing environment

The testing environment used in this test is a room temperature of 20 °C and a relative humidity of 65 % in accordance with ISO 139.

## 7 Test procedure

### 7.1 General

Two preparation methods are described in this clause as method A and method B. One method shall be selected with the consent of the concerned parties.

### 7.2 Preparation of specimen

The dimension or mass of specimen for the textile products is shown in [Table 1](#).

**Table 1 — Dimension or mass of specimen**

Kind of specimen	Dimension or mass of specimen	
	Method A	Method B
Fabrics (woven, knit, nonwoven, and tapes)	50 cm <sup>2</sup> ± 2,5 cm <sup>2</sup>	25 cm <sup>2</sup> ± 1,25 cm <sup>2</sup>
Yarns, fibres, and feather	0,5 g ± 0,025 g	—

NOTE In the case of multi-layer products, the edge and non-treated layer (or not concerned layer) can be covered with aluminium sheets to avoid contact with the odorous atmosphere, or the product can be folded in two with the layer that is not concerned inside, in method A.

### 7.3 Method A

7.3.1 Prepare six conical flasks (5.1) with 500 ml: three conical flasks are used for testing with a specimen and three conical flasks are used for testing without a specimen as a control test.

7.3.2 Clean the flasks by using nitrogen gas or clean air more than five times of volume of the flask.

1) Parafilm is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.



### 7.3.3 Preparation of the odour component chemical solution.

The test is performed for each odour component chemical independently.

Prepare the odour component chemical solutions for the test separately as the following.

#### 7.3.3.1 Indole

Dissolve 20 g of the indole reagent (4.2) into 1 l of ethanol (4.7).

#### 7.3.3.2 Isovaleric acid

Dissolve 20 g of isovaleric acid reagent (4.3) into 1 l of ethanol (4.7).

#### 7.3.3.3 Nonenal

Dissolve 10 g of nonenal reagent (4.4) into 1 l of ethanol (4.7).

### 7.3.4 Testing with specimen

#### 7.3.4.1 Preparation of specimen

Take three specimens from the sample for one chemical test as shown in [Table 1](#).

#### 7.3.4.2 Placement of specimen

Put each specimen in a conical flask (5.1) to lie flat on the bottom.

#### 7.3.4.3 Nitrogen purge

Purge the air of the conical flask by blowing of 1 000 ml of nitrogen gas.

Seal the mouth of the conical flask by using sealing film (5.2).

#### 7.3.4.4 Insertion of testing odour component chemical solution

Take 5 µl of the odour component chemical solution prepared in [7.3.3.1](#), [7.3.3.2](#), or [7.3.3.3](#), by using the injection syringe (5.4) through the sealing film and inject the testing solution into the conical flask at the bottom edge of the conical flask, avoiding contact with the specimen.

#### 7.3.4.5 Sealing of the microsyringe hole

Seal the hole of the microsyringe by the sealing film (5.2) over the original seal.

#### 7.3.4.6 Contacting time

Keep the conical flask still for 2 h, without agitation or stirring of the testing gas.

### 7.3.5 Testing without specimen

For testing without a specimen, repeat the procedure from [7.3.4.2](#) to [7.3.4.6](#) without a specimen.

### 7.3.6 Sampling of the testing gas

**7.3.6.1** After 2 h of contacting time, hold the mouth part of the conical flask and shake approximately 20 times for 20 s vigorously for both flasks of testing gases with and without a specimen.

**7.3.6.2** Insert a gastight syringe (5.5) vertically into the centre of the sealing film of the mouth of the conical flask and insert the needle by approximately 4 cm inside the conical flask.

**7.3.6.3** Take the testing gas from inside the conical flask by the gastight syringe.

The amount of sampling gas depends on the model of the gas chromatography apparatus used.

NOTE When the gastight syringe is used repeatedly, the repeated suction and discharge is recommended for several times to avoid the influence of residual gases in the syringe.

## 7.4 Method B

### 7.4.1 Preparation of vials

**7.4.1.1** Prepare six injection vials (5.3) with 22 ml: three vials are used for testing with a specimen and three vials are used for testing without a specimen for a control test.

**7.4.1.2** Clean the vials by using nitrogen gas (4.5) or clean air more than five times of the volume of the vial.

### 7.4.2 Preparation of acetic acid and NaCl solution

Prepare acetic acid and NaCl solution with 1 g/l of acetic acid (4.1) and 9 g/l of NaCl (4.6) in 1 l of water.

### 7.4.3 Preparation of specimen

Take three specimens from the sample for one test and make a roll respectively.

### 7.4.4 Testing with specimen

#### 7.4.4.1 Placement of specimen

Place each roll of specimen vertically in the three injection vials and seal the injection vials with rubber stoppers and aluminium caps (5.8).

#### 7.4.4.2 Injection of the acetic acid and NaCl solution

Take 850 µl of the acetic acid and NaCl solution with the injection syringe for each test and inject on to the fabric directly for three vials with a specimen through the rubber stoppers (5.8) by using the injection syringe (5.5).

#### 7.4.4.3 Contacting time

Place the six vials with the following sequence:

- vial with specimen;
- vial without specimen;
- vial with specimen;
- vial without specimen;
- vial with specimen;
- vial without specimen.

Place them in the heating oven at a temperature of 80 °C ± 2 °C for 30 min ± 3 min.

### 7.4.5 Testing without specimen

Repeat the procedure in 7.4.4.1 to 7.4.4.3 without a specimen.

#### 7.4.6 Sampling of the odour testing gas

Take 1 ml of odour testing gas after contacting time from each vial, with or without a specimen, by using the injection syringe (5.5).

### 7.5 Concentration measurement of testing gas by GC

#### 7.5.1 General

The testing gas obtained in 7.3.5 or 7.4.6 is injected into the column of the gas chromatograph, and the concentration of the odour component chemical is detected by a hydrogen flame ionization detector (FID). The peak area value of the FID spectrum is obtained as the value proportional to the chemical concentration of the testing gas.

#### 7.5.2 Peak area of FID spectrum of testing gas with specimen

Measure the peak area of the FID spectrum for three testing gases with a specimen by GC and take an average which is denoted as  $S_m$ .

#### 7.5.3 Concentration of testing gas without specimen

Measure the peak area of the FID chromatogram for three testing gases without a specimen by GC and take an average which is denoted as  $S_b$ .

## 8 Calculation of odour reduction rate

Calculate the odour reduction rate according to the Formula (1).

$$ORR = \frac{(S_b - S_m)}{S_b} \times 100 \quad (1)$$

where

$ORR$  is the odour reduction rate, as a percentage;

$S_m$  is the average peak area of FID of the testing gas with a specimen;

$S_b$  is the average peak area of FID spectrum of the testing gas without a specimen.

## Annex A (informative)

### GC parameter

Capillary column: DB-5 [Agilent J&W<sup>2</sup>], length: 30 m, inside diameter: 0,53 mm

Film thickness: 1,0 µm

Injector system: split or splitless

Injector temperature: 250 °C

Carrier gas: helium

Temperature programme: 120 °C (constant for nonenal and indole), 70 °C (constant for isovaleric acid)

Injection volume: 1,0 ml

Detection: FID

---

2) Agilent J&W is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

## Annex B (informative)

### Round-robin test result

#### B.1 Round-robin test

##### B.1.1 General

The odour reduction rate (*ORR*) % was calculated in [Tables B.1](#) to [B.4](#) from the data of the peak area of FID spectrum, with and without a specimen, by using method A.

##### B.1.2 Samples

Polyester woven fabric:

- B-1 treated by a low concentration of deodorant substance;
- B-2 treated by a medium concentration of deodorant substance;
- B-3 treated by a high concentration of deodorant substance.

##### B.1.3 Tested odour chemical gases

- Isovaleric acid
- Nonenal

#### B.2 Test result

##### B.2.1 Isovaleric acid

###### B.2.1.1 Testing gas peak area of FID spectrum without a specimen

**Table B.1 — Isovaleric acid testing gas peak area without a specimen**

Testing laboratory	Peak area without a specimen				
	<i>n</i> = 1	<i>n</i> = 2	<i>n</i> = 3	Mean $S_b$	STD
A	72 126	71 236	72 879	72 080	822
B	104 694	119 187	96 260	106 714	11 596
C	46 951	52 161		49 556	3 684
D	528 459	403 396	415 600	449 152	68 953
E	6 874	6 954	9 363	7 730	1 414
F	70 420	61 609	50 627	60 885	9 916

###### B.2.1.2 Testing gas peak area of FID spectrum with specimen B-1 and *ORR* %

*ORR* % is the reduction rate which is calculated by using data from [Tables B.1](#), [B.2](#), [B.3](#), and [B.4](#).

**Table B.2 — Isovaleric testing gas peak area with specimen B-1 and *ORR* %**

Testing laboratory	With the specimen B-1					
	Peak area					<i>ORR</i> %
	<i>n</i> = 1	<i>n</i> = 2	<i>n</i> = 3	Mean $S_m$	STD	
A	40 091	38 270		39 181	1 288	45,6
B	59 738	60 480	57 467	59 228	1 570	44,5
C	37 806	34 122		35 964	2 605	27,4
D	317 345	233 397	314 013	288 252	47 535	35,8
E	4 692	5 093	4 690	4 825	232	37,6
F	29 759	47 152	42 656	39 856	9 028	34,5
Mean						37,6 %
STD (standard deviation)						6,8 %

**Table B.3 — Isovaleric acid testing gas peak area with specimen B-2 and *ORR* %**

Testing laboratory	With the specimen B-2					
	Peak area					<i>ORR</i> %
	<i>n</i> = 1	<i>n</i> = 2	<i>n</i> = 3	Mean $S_m$	STD	
A	18 678	17 753		18 216	654	74,7
B	21 760	25 134	22 624	23 173	1 753	78,3
C	13 526	14 639		14 083	787	71,6
D	131 928	148 631	149 447	143 335	9 887	68,1
E	2 658	1 946	2 482	2 362	371	69,4
F	15 049	8 942	13 214	12 402	3 133	79,6
Mean						73,6 %
STD						4,7 %

**Table B.4 — Isovaleric acid testing gas peak area with specimen B-3 and *ORR* %**

Testing laboratory	With the specimen B-3					
	Peak area					<i>ORR</i> %
	<i>n</i> = 1	<i>n</i> = 2	<i>n</i> = 3	Mean $S_m$	STD	
A	5 415	4 888		5 152	373	92,9
B	12 554	9 859	12 093	11 502	1 441	89,2
C	6 020	4 757		5 389	893	89,1
D	77 878	71 864	70 236	73 326	4 025	83,7
E	675	1 067	673	805	227	89,6
F	6 014	6 761	8 039	6 938	1 024	88,6
Mean						88,8 %
STD						3,0 %

## B.2.2 Nonenal

### B.2.2.1 Testing gas peak area of FID spectrum without specimen

**Table B.5 — Nonenal testing gas peak area without specimen**

Testing laboratory	Without-specimen peak area				
	<i>n</i> = 1	<i>n</i> = 2	<i>n</i> = 3	Mean $S_b$	STD
A	126 192	140 473	139 504	135 390	7 980
B	62 654	60 752	64 023	62 476	1 643
C	17 313	18 097	18 961	18 124	824
D	123 996	132 853	132 963	129 937	5 146
E	40 412	43 557	29 935	37 968	7 132
F	67 373	64 031	86 967	72 790	12 391

### B.2.2.2 Testing gas peak area of FID spectrum with specimens

**Table B.6 — Nonenal testing gas peak area with specimen B-1 and ORR %**

Testing laboratory	With the specimen B-1					ORR %
	Peak area					
	<i>n</i> = 1	<i>n</i> = 2	<i>n</i> = 3	Mean $S_m$	STD	
A	56 752	59 570	53 820	56 714	2 875	58,1
B	21 307	20 539	22 646	21 497	1 066	65,6
C	6 759	6 550	6 514	6 608	132	63,5
D	58 474	57 387	56 127	57 329	1 175	55,9
E	15 651	13 074	14 093	14 273	1 298	62,4
F	31 801	26 290	23 649	27 247	4 159	62,6
Mean						61,3 %
STD						3,6 %

**Table B.7 — Nonenal testing gas peak area with specimen B-2 and ORR %**

Testing laboratory	With the specimen B-2					ORR %
	Peak area					
	<i>n</i> = 1	<i>n</i> = 2	<i>n</i> = 3	Mean $S_m$	STD	
A	24 321	29 846	21 600	25 256	4 202	81,3
B	8 541	7 009	7 227	7 592	829	87,8
C	3 008	2 805	3 282	3 032	239	83,3
D	30 509	34 059	32 601	32 390	1 784	75,1
E	7 450	7 534	6 729	7 238	443	80,9
F	15 490	12 710	11 020	13 073	2 257	82,0
Mean						81,7
STD						4,1

Table B.8 — Nonenal testing gas peak area with specimen B-3 and *ORR* %

Testing laboratory	With the specimen B-3					
	Peak area					<i>ORR</i> %
	<i>n</i> = 1	<i>n</i> = 2	<i>n</i> = 3	Mean $S_m$	STD	
A	9 095	12 776	9 392	10 421	2 045	92,3
B	3 870	3 875	3 572	3 772	174	94,0
C	1 511	1 411	1 369	1 430	73	92,1
D	14 974	14 057	13 896	14 309	582	89,0
E	2 781	2 264	2 378	2 474	272	93,5
F	8 248	6 383	7 135	7 255	938	90,0
Mean						91,8 %
STD						2,0 %

### B.2.3 Summary of test result

Table B.9 — Summary of the test result

Sample	ID code	B-1	B-2	B-3
	Deodorant treatment concentration	Low	Medium	High
Isovaleric acid	<i>ORR</i> (%)	37,6	73,6	88,8
	STD (%)	6,8	4,7	3,0
Nonenal	<i>ORR</i> (%)	61,3	81,7	91,8
	STD (%)	3,6	4,1	2,0





.....