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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

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Foreword

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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_3COOH)**, reagent with a purity of 99,7 %.

4.2 **Indole (C_8H_7N)**, reagent.

4.3 **Isovaleric acid**, reagent with a purity of 98,0 %.

4.4 **2-Nonenal ($C_9H_{16}O$)**, 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[1)]].

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^2 ± 2,5 cm^2	25 cm^2 ± 1,25 cm^2
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.

7.4.12 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[2)]], 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				
	$n = 1$	$n = 2$	$n = 3$	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					*ORR* %
	$n = 1$	$n = 2$	$n = 3$	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					*ORR* %
	$n = 1$	$n = 2$	$n = 3$	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					*ORR* %
	$n = 1$	$n = 2$	$n = 3$	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 %