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Textiles — Determination of deodorant property —

Part 2: Detector tube method

Textiles — Détermination des propriétés de neutralisation d'odeurs —

Partie 2: Méthode du tube détecteur

Reference number
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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.

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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 method using a detector tube as a concentration measuring device. The detector tube is a well-known odour sensor used for measurement of environmental odour chemical concentration in the field. The detector tube is made for each specific chemical and could be used for concentration measurement for a targeted chemical in gases. Detector tubes could be available commercially. This is a very simple and inexpensive testing method if the detector tubes can be obtained.

Textiles — Determination of deodorant property —

Part 2: Detector tube method

1 Scope

This part of ISO 17299 specifies a deodorant testing method using a detector tube for all textile products. This method is applicable to the odour component chemicals of ammonia, acetic acid, methyl mercaptan, and hydrogen sulfide.

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 17299-1, *Textiles — Determination of deodorant property — Part 1: General principle*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
detector tube
device used for the gas concentration measurement test, which is a glass tube filled by grainy chemicals which react to the odour chemicals and change colour in proportion with the concentration of testing chemical

Note 1 to entry: The concentration of chemicals can be read on the graduation printed on the surface of the glass tube.

4 Principle

Concentration of gaseous odour component chemicals of gas in containers with or without a test specimen after a designated contacting time is measured by using detector tubes. The odour reduction rate (*ORR*) % of chemical concentration is calculated from the concentration data with a specimen and without a specimen. Each chemical shall be tested individually.

5 Reagents

5.1 **Ammonia water (NH_3)**, reagent with a concentration of 28 % in water.

5.2 **Acetic acid (CH_3COOH)**, reagent with a purity of 99,7 %.

5.3 **Methyl mercaptan (CH_3SH)**, standard gas with a concentration of 100 µl/l or 1 000 µl/l by nitrogen dilution.

5.4 **Hydrogen sulfide (H_2S)**, standard gas with a concentration of 100 µl/l by nitrogen dilution.

5.5 Diluent gas, dry air obtained from the mixture cylinder of nitrogen gas and oxygen gas with a purity of at least 99,99 %, or nitrogen gas from the nitrogen gas cylinder with a purity of at least 99,99 %.

6 Materials and apparatus

6.1 Detector tube, the measuring ranges of the tubes are given as the following with an accuracy of ±5 %.

— for ammonia: 0,2 µl/l to 200 µl/l;

— for acetic acid: 0,25 µl/l to 50 µl/l;

— for methyl mercaptan: 0,5 µl/l to 10 µl/l;

— for hydrogen sulfide: 0,2 µl/l to 6 µl/l.

NOTE Follow the manufacturer's instruction for the usable time and condition for storage which are indicated on the detector tube.

6.2 Plastic bag, with a volume of 1 l, 5 l, and 50 l, made of vinyl fluoride film, polyester, polyester laminated film, polyvinyl alcohol film, etc. A plastic or rubber tube is installed to the bag before testing.

6.3 Air pump, capable of drawing air with a flow rate of 0,2 l/min and 5 l/min with the attached flow meter. If the attached flow meter is not available, the integrating flow meter shall be used.

6.4 Integrating flow meter, capable of measuring the gas flow of at least 500 ml/min.

6.5 Syringe, made of a glass cylinder with a capacity of 0,5 ml and 100 ml.

6.6 Airtight stopper.

7 Preparation

7.1 Preparation of the testing gas

The test for each odour component chemical is performed separately.

Prepare each odour component chemical just before the test.

NOTE Other gas preparation methods, such as use of standard gas generation instrument (e.g. parmeator or cylinder) are also usable.

7.1.1 Ammonia

7.1.1.1 Preparation of ammonia gas

7.1.1.1.1 Prepare the 1 l plastic bag (6.2), well-cleaned just before testing.

7.1.1.1.2 Insert 500 ml of the diluent gas (5.5) by using the air pump (6.3) and attach an airtight stopper (6.6).

NOTE If no flow meter is attached, the integrating flow meter (6.4) is available to measure the gas flow.

7.1.1.1.3 Take 0,5 ml of ammonia water (5.1) with the syringe (6.5) and inject it into the plastic bag.

7.1.1.1.4 Warm ammonia water to a temperature of approximately 40 °C to 50 °C for about 3 min with a hand dryer or oven to vaporize the ammonia water. Mix the ammonia gas well by kneading the plastic bag.

NOTE The concentration of ammonia gas is approximately 160 000 µl/l.

7.1.1.1.5 Take 65 ml of the ammonia gas prepared in 7.1.1.1.4 by using the 100 ml syringe (6.5) and insert the gas into a new 1 l plastic bag which was cleaned well by diluent gas and deaerated beforehand.

7.1.1.1.6 Insert the dilution gas to make up a total volume of 1 l. Mix the gas well by kneading the plastic bag.

NOTE The concentration of ammonia gas is approximately 10 400 µl/l.

7.1.1.1.7 Take 250 ml of the ammonia gas prepared in 7.1.1.1.6 by using the 100 ml syringe (6.5) three times and insert the gas into a new 50 l plastic bag (6.2) which is cleaned well by diluent gas (5.5) and deaerated beforehand.

7.1.1.1.8 Insert the dilution gas to make up a total volume of 25 l. This becomes a master gas with the concentration of 100 µl/l.

NOTE A smaller size of plastic bag is available instead of 50 l.

7.1.1.2 Confirmation of ammonia master gas concentration

7.1.1.2.1 Insert 3 l of the master gas into a 5 l plastic bag by using an air pump (6.3). Take the 100 ml gas sample from the bag by using the 100 ml syringe. Measure the gas concentration by using the detector tube.

7.1.1.2.2 Confirm that the gas concentration is in the range of 100 µl/l ± 5 µl/l.

NOTE If the gas concentration is outside the limit, do not use the gas as the master gas. Prepare it again.

7.1.2 Acetic acid

Prepare the master gas with 30 µl/l ± 3 µl/l of acetic acid concentration by using acetic acid reagent (5.2) with 99,7 % concentration of water solution and by using diluent gas (5.5).

7.1.3 Methyl mercaptan

Prepare the master gas with 8 µl/l ± 0,8 µl/l of methyl mercaptan concentration by using the standard methyl mercaptan gas (5.3) and by using diluent gas (5.5).

7.1.4 Hydrogen sulfide

Prepare the master gas with 4 µl/l ± 0,4 µl/l of hydrogen sulfide concentration by using the standard hydrogen sulfide gas (5.4) with 100 µl/l concentration in nitrogen gas and by using diluent gas (5.5).

8 Testing environment

The testing environment shall be kept at a temperature of 20 °C and relative humidity of 65 % in accordance with ISO 139.

9 Preparation of specimen

9.1 The dimension or mass of specimens is shown in Table 1. The number of test specimens is 3 for one test.

Table 1 — Dimension or mass of specimen

Kind of sample	Dimension or mass of specimen
Fabrics (woven, knit, nonwoven) and tapes	100 cm^2 ± 5 cm^2
Yarns, braid, fibres, and feather	1,0 g ± 0,05 g

NOTE In the case of multi-layer products, the edge and non-treated layer (or not concerned layer) can be covered with aluminium foil to avoid contact with odorous gas, or the specimen can be folded in two as with the layer that is not concerned inside.

9.2 Condition the samples under the same conditions as for testing for at least 24 h.

10 Test procedure

10.1 Prepare six plastic bags with a volume of 5 l.

10.2 Test with a specimen.

10.2.1 Insert the specimen as follows.

10.2.1.1 Place the specimen in the three plastic bags, one by one and spread it as much as possible.

10.2.1.2 Seal the plastic bag in which the specimen was placed for testing, by using a heat seal or seal tape.

NOTE Curling and creasing of the specimen might be avoidable.

10.2.2 Deaerate from the bag as much as possible by an aspirator or a vacuum pump.

NOTE It is easy to deaerate if the specimen is placed near the mouth of the plastic bag.

10.2.3 Insert 3 l of the odour component testing gas into the plastic bags by using an air pump.

10.2.4 Place the plastic bag in a still condition for 2 h for contacting.

10.2.5 Extract 100 ml of the testing gas from the three plastic bags with specimen by using the 100 ml syringe.

10.2.6 Pass the extracted testing gas through the detector tube; then, read the scale at the discoloration point.

This value represents the concentration of odour component chemicals after contacting time with the specimen.

10.2.7 Take an average of three odour gas concentration data with a specimen, which is denoted as *A*.

10.3 Test without a specimen

10.3.1 This is a control test with the same testing conditions as 10.2.2 to 10.2.6 except without a specimen.

10.3.2 Take an average of three odour gas concentration data without a specimen, which is denoted as *B*.

11 Odour reduction rate calculation

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

$$ORR = \frac{(B - A)}{B} \times 100 \quad (1)$$

where

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

B is the average of the concentration of testing gas without a specimen, expressed in µl/l;

A is the average of the concentration of testing gas with a specimen, expressed in µl/l.

Annex A
(informative)

Round-robin test result

A.1 Round-robin test

A.1.1 General

The odour reduction rate (*ORR*) was calculated in Tables A.1 to A.4 from the data of the chemical concentration with and without a specimen.

A.1.2 Samples

Polyester woven fabric:

— A-1 treated by a low concentration of deodorant substance;

— A-2 treated by a medium concentration of deodorant substance;

— A-3 treated by a high concentration of deodorant substance.

A.1.3 Tested odour chemical gases

— Ammonia

— Acetic acid

A.2 Test result

A.2.1 Ammonia gas

A.2.1.1 Testing gas concentration without a specimen

Table A.1 — Ammonia testing gas concentration without a specimen

Testing laboratory	Without specimen concentration µl/l				
	$n = 1$	$n = 2$	$n = 3$	Mean B	Variance
A	70	70	69	69,7	0,3
B	82	81	79	80,7	2,3
C	95	95	95	95,0	0,0
D	81	83	83	82,3	1,3
E	94	94	93	93,7	0,3
F	74	73	75	74,0	1,0
Mean				82,6	
Repeatability variance (average)					0,9
Between-laboratory variance					104,3

Table A.1 *(continued)*

Testing laboratory	Without specimen concentration µl/l				
	$n = 1$	$n = 2$	$n = 3$	Mean B	Variance
Reproducibility variance					105,1
STD (standard deviation) of repeatability					0,94
STD of reproducibility					10,3
CV (coefficient of variation) % of repeatability					1,1 %
CV % of reproducibility					12,4 %

A.2.1.2 Testing gas concentration with specimens

ORR % is the reduction rate which is calculated by using data from Tables A.1, A.2, A.3, and A.4.

Table A.2 — Ammonia testing gas concentration with specimen A-1 and *ORR*

Testing laboratory	With the specimen A-1					
	Concentration µl/l				Variance	*ORR* %
	$n = 1$	$n = 2$	$n = 3$	Mean A		
A	38	37	37	37,3	0,3	46,4
B	42	37	42	40,3	8,3	50,0
C	55	54	52	53,7	2,3	43,5
D	43	44	40	42,3	4,3	48,6
E	52	50	55	52,3	6,3	44,1
F	44	42	38	41,3	9,3	44,1
Mean				44,6		46,1
Repeatability variance					5,17	
Between- laboratory variance					45,8	
Reproducibility variance					50,9	
STD of repeatability					2,27	
STD of reproducibility					7,1	2,7 %
CV % of repeatability					5,1 %	
CV % of reproducibility					16,0 %	5,8 %

Table A.3 — Ammonia testing gas concentration with specimen A-2 and *ORR*

Testing laboratory	With the specimen A-2					
	Concentration µl/l				Variance	*ORR* %
	$n = 1$	$n = 2$	$n = 3$	Mean A		
A	25	24	24	24,3	0,3	65,1
B	27	23	24	24,7	4,3	69,4
C	35	35	38	36,0	3,0	62,1

Table A.3 *(continued)*

Testing laboratory	With the specimen A-2					
	Concentration µl/l				Variance	*ORR* %
	n = 1	*n* = 2	*n* = 3	Mean *A*		
D	20	20	21	20,3	0,3	75,3
E	35	38	33	35,3	6,3	62,3
F	23	19	22	21,3	4,3	71,2
Mean				27		67,6
Repeatability variance					3,1	
Between-laboratory variance					47,9	
Reproducibility variance					51,0	
STD of repeatability					1,76	
STD of reproducibility					7,1	5,2 %
CV % of repeatability					6,5 %	
CV % of reproducibility					26,5 %	7,8 %

Table A.4 — Ammonia testing gas concentration with specimen A-3 and *ORR*

Testing laboratory	With the specimen A-3					
	Concentration µl/l				Variance	*ORR* %
	n = 1	*n* = 2	*n* = 3	Mean *A*		
A	15	14	15	14,7	0,3	78,9
B	16	14	15	15,0	1,0	81,4
C	22	25	20	22,3	6,3	76,5
D	12	14	12	12,7	1,3	84,6
E	20	20	18	19,3	1,3	79,4
F	13	12	11	12,0	1,0	83,8
Mean				16,0		80,8
Repeatability variance					1,89	
Between-laboratory variance					16,2	
Reproducibility variance					18,1	
STD of repeatability					1,37	
STD of reproducibility					4,3	3,1 %
CV % of repeatability					8,6 %	
CV % of reproducibility					26,6 %	3,8 %

A.2.2 Acetic acid gas

A.2.2.1 Testing gas concentration without a specimen

Table A.5 — Acetic acid testing gas concentration without a specimen

Testing laboratory	Without-specimen concentration µl/l				
	$n = 1$	$n = 2$	$n = 3$	Mean B	Variance
A	20	22	22	21,3	1,3
B	24	25	27	25,3	2,3
C	33	32,6	31,6	32,4	0,5
D	32	32	31	31,7	0,3
E	23	24	24	23,7	0,3
F	31	31	31	31,0	0,0
Mean				27,6	
Repeatability variance					0,81
Between-laboratory variance					22,2
Reproducibility variance					23,0
STD of repeatability					0,90
STD of reproducibility					4,8
CV % of repeatability					3,3 %
CV % of reproducibility					17,4 %

A.2.2.2 Testing gas concentration with specimens

Table A.6 — Acetic acid testing gas concentration with specimen A-1 and *ORR*

Testing laboratory	With the specimen A-1					
	Concentration µl/l					*ORR* %
	$n = 1$	$n = 2$	$n = 3$	Mean A	Variance	
A	5,1	5,1	4,6	4,9	0,08	76,9
B	9,5	11	11	10,5	0,75	58,6
C	10	10	10	10,0	0,00	69,1
D	14	15	15	14,7	0,33	53,7
E	6	6	5,5	5,8	0,08	75,4
F	13	13	12,5	12,8	0,08	58,6
Mean				9,78		65,4
Repeatability variance					0,22	
Between-laboratory variance					14,7	
Reproducibility variance					14,9	
STD of repeatability					0,47	
STD of reproducibility					3,9	9,7
CV % of repeatability					4,8 %	
CV % of reproducibility					39,9 %	14,9 %

Table A.7 — Acetic acid testing gas concentration with specimen A-2 and *ORR*

Testing laboratory	With the specimen A-2					
	Concentration µl/l					*ORR* %
	n = 1	*n* = 2	*n* = 3	Mean *A*	Variance	
A	2,3	4,1	3,6	3,3	0,86	84,4
B	7	8,5	7	7,5	0,75	70,4
C	8	8	8	8,0	0,00	75,3
D	10	10	10	10,0	0,0	68,4
E	4	4	3,5	3,8	0,08	83,8
F	10,5	11	11	10,8	0,08	65,1
Mean				7,3		74,6
Repeatability variance					0,3	
Between-laboratory variance					9,7	
Reproducibility variance					10	
STD of repeatability					0,54	
STD of reproducibility					3,2	8,1
CV % of repeatability					7,4 %	
CV % of reproducibility					43,2 %	10,9 %

Table A.8 — Acetic acid testing gas concentration with specimen A-3 and *ORR*

Testing laboratory	With the specimen A-3					
	Concentration µl/l					*ORR* %
	n = 1	*n* = 2	*n* = 3	Mean *A*	Variance	
A	2,3	1,8	1,5	1,9	0,16	91,1
B	4,5	5	5	4,8	0,08	81,0
C	5	5	5	5,0	0,0	84,6
D	6	7	5,5	6,2	0,58	80,4
E	2	2,5	2,5	2,3	0,08	90,2
F	6,9	7,1	7,1	7,0	0,01	77,4
Mean				4,5		84,1
Repeatability variance					0,15	
Between-laboratory variance					4,2	
Reproducibility variance					4,4	
STD of repeatability					0,39	
STD of reproducibility					2,1	5,6
CV % of repeatability					8,7%	
CV % of reproducibility					46,7 %	6,7 %

A.2.3 Summary of result

Table A.9 — Summary of the result

Sample	Code	A-1	A-2	A-3
	Deodorant treatment concentration	Low	Medium	High
Ammonia	*ORR* (%)	46,1	67,6	80,8
	STD (%)	2,7	5,2	3,1
Acetic acid	*ORR* (%)	65,4	74,6	84,1
	STD (%)	9,7	8,1	5,6

A.2.4 Interpretation of result

For the interpretation of results, use ISO 17299-1.

Annex B
(informative)

Detector tube suppliers

B.1 Supplier information

NOTE This information is given for the convenience of users of this part of ISO 17299 and does not constitute an endorsement by ISO of these detector tubes.

a) Gastec: http://www.gastec.co.jp/english/index.php

b) Komyo Rikagaku Kogyo K.K.: http://www.komyokk.co.jp/kweb/top_page.do

c) Draeger: http://www.draeger.jp/MT/internet/JP/jp/prodserv/academy/int_academy_top.jsp

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