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Textiles — Determination of deodorant property —

Part 5: Metal-oxide semiconductor sensor method

Textiles — Détermination de la propriété de déodorant —
Partie 5: Méthode par capteur à semi-conducteur métal-oxyde

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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

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Foreword

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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 sensor method*

Introduction

This part of ISO 17299 describes a test method using a testing instrument equipped with multiple metal-oxide semiconductor sensors against composite odours for all textiles. The multiple sensors improve accuracy for several kinds of composite odours.

Textiles — Determination of deodorant property —

Part 5: Metal-oxide semiconductor sensor method

1 Scope

This part of ISO 17299 specifies a deodorant test method using an odour analyser equipped with multiple metal-oxide semiconductor sensors against composite odours. The artificial composite odours used in this part of ISO 17299 are a quasi sweat odour, a quasi body odour (nonenal mixture odour) and a quasi excrement odour.

This part of ISO 17299 applies to all kinds of textile products, such as woven fabrics, knits, threads, yarns, fibres, braids, cords, etc.

2 Normative references

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
quasi unpleasant odour
artificial composite odour used to simulate an unpleasant odour

Note 1 to entry: Artificial composite odours for the purpose of this part of ISO 17299 are as follows:

— quasi sweat odour: ammonia, acetic acid, isovaleric acid;

— quasi body odour (nonenal mixture odour): ammonia, acetic acid, isovaleric acid, nonenal;

— quasi excrement odour: ammonia, acetic acid, hydrogen sulfide, methyl mercaptan, indole.

3.2
odour unit concentration
concentration defined as the rate of the odour chemical concentration in µl/l against the olfactory threshold concentration

Note 1 to entry: It is expressed in OU/m^3.

3.3
olfactory threshold concentration
minimum concentration at which human could perceive

Note 1 to entry: Olfactory threshold concentration value of the odour chemical substances used in this part of ISO 17299 is shown in Table 1.

Note 2 to entry: See Reference [1] in the Bibliography.

Table 1 — Olfactory threshold concentration

Unpleasant odour components	Ammonia	Acetic acid	Isovaleric aicid	Nonenal	Hydrogen sulfide	Methyl mercaptan	Indole
Olfactory threshold concentration [µl/l]	1,5	0,006	0,000 078	0,000 08	0,000 41	0,000 07	0,000 3

3.4
metal-oxide semiconductor sensor
sensor using a metal-oxide semiconductor made of e.g. tin dioxide

Note 1 to entry: The response of sensors corresponds to gas chemical concentration. The blend recipe of the constituent metal-oxide gives different response trend to chemicals.

3.5
odour test instrument
instrument equipped with multiple metal-oxide semiconductor sensors

Note 1 to entry: This instrument is built up with the inlet of the testing gas, sensors, and cleaning system of sensor head.

3.6
response value
value obtained as outputs from sensors

3.7
response vector
vector for a quasi unpleasant odour consisting of response value obtained from each sensor separately

3.8
response vector length
length obtained as vector sum which is calculated as square-root of sum of squares of response values

Note 1 to entry: The length represents a proportional amount of chemical concentration.

3.9
calibration curve
curve that determines odour unit concentration corresponding to response vector length

Note 1 to entry: The quadratic curve is used for this calibration curve derived from Reference [2] as proximity.

4 Principle

The quasi unpleasant odour gas is inserted in plastic bags with test specimen or without specimen. After 2 h reaction time, the odour unit concentration of remained gases is obtained by using an odour test instrument with 10 metal-oxide semiconductor sensors. The reduction rate in odour chemical concentration is calculated from the odour unit concentration with and without specimen.

5 Reagents

Unless otherwise specified, analytical grade reagents shall be used in this test.

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

5.2 **Acetic acid**, reagent of 99,7 % in concentration of acetic acid (CH_3COOH) in water.

5.3 **Methyl mercaptan**, 100 µl/l in concentration of standard gas by nitrogen dilution.

5.4 **Hydrogen sulfide**, 100 µl/l in concentration of standard gas by nitrogen dilution.

5.5 **Indole**, indole (C_8H_7N) reagent.

5.6 **Isovaleric acid**, 98,0 % solution.

5.7 **Nonenal**, 2-nonenal ($C_9H_{16}O$) reagent (95,0 % solution).

5.8 **Diluent gas**, dry air obtained from nitrogen - oxygen mixture cylinder with purity of at least 99,999 9 % or, nitrogen gas from nitrogen gas cylinder with purity of at least 99,999 9 %.

6 Apparatus and materials

Unless otherwise specified, the following shall be used in this test.

6.1 **Plastic bag**, 3 l in capacity with a glass tube as inlet of gas.

6.2 **Air pump**, with a flow meter or an integrating flow meter for 3 l/min flow rate.

6.3 **Oven**, capable of operating at 80 °C.

6.4 **Micro-syringe**, with a capacity of 10 µl.

6.5 **Syringe**, with a capacity of 200 ml.

6.6 **Detector tube**, with a concentration measurement range of 50 µl/l to 500 µl/l for ammonia.

6.7 **Heat seal**, capable of sealing a plastic bag.

6.8 **Aspirator** or **vacuum pump**.

6.9 **Odour test instrument**, with 10 metal-oxide semiconductor sensors.

7 Preparation of the odour test gas

The odour test gases are prepared just before the test. Master gases and the quasi unpleasant odours may be prepared by permeator or gas cylinder.

7.1 Master gas

7.1.1 Clean 3 l of plastic bags (6.1) by using the diluent gas, then the preparation of master gases of odour component chemicals is as described in 7.1.2 to 7.1.4.

7.1.2 Ammonia, acetic acid, isovaleric acid, and nonenal

7.1.2.1 Inject 2,5 l of the diluent gas into the plastic bags (7.1.1).

7.1.2.2 Inject the chemical solution with the amount according to Table 2 into plastic bags prepared in 7.1.2.1.

7.1.2.3 Keep the plastic bags with the prepared gas under the test environment for 30 min.

7.1.3 Hydrogen sulfide and methyl mercaptan

7.1.3.1 Inject the chemical standard gases with the amount according to Table 2 into plastic bags.

7.1.4 Indole

7.1.4.1 Put the specified amount of indole powder into a plastic bag (6.1) according to Table 2.

7.1.4.2 Inject 2,5 l of dilute gas in the plastic bag.

7.1.4.3 Heat the plastic bag for sublimation at 80 °C for 10 min.

7.1.4.4 Transfer all gas of 7.1.4.3 to new plastic bag.

Table 2 — Master gas concentration of unpleasant odour components and manufacture method

Master gas	Ammonia	Acetic acid	Isovaleric acid	Nonenal	Hydrogen sulfide	Methyl mercaptan	Indole
Master gas target concentration [µl/l]	1 500	500	50	15	100	100	6
Diluent gas quantity [ml]	2 500	2 500	2 500	2 500	0	0	2 500
Materials of odour components chemicals	Ammonia water (5.1)	Acetic acid (5.2)	Isovaleric acid (5.6)	Nonenal (5.7)	Hydrogen sulfide (5.4)	Methyl mercaptan (5.3)	Indole reagent (5.5)
Quantity of injection odour component chemicals	10 µl	5 µl	2 µl	5 µl	2 500 ml	2 500 ml	0,3 g powder
Inspection	Dilute to 1/10 and use detector tube	No action					

7.2 Preparation of the test quasi unpleasant odours

The compositions of the quasi unpleasant odours are shown in Table 3. The compositions give the initial concentration of odour chemicals for test.

Table 3 — Composition of chemicals in the quasi unpleasant odours

Unpleasant odour components		Composition of chemicals for master gas (ml)								Total (ml)
		Ammonia	Acetic acid	Isovaleric acid	Nonenal	Hydrogen sulfide	Methyl mercaptan	Indole	Diluent gas	
Quasi unpleasant odour	Sweat odour	50	250	500	-	-	-	-	1 700	2 500
	Body odour (nonenal mixture odour)	50	250	500	830	-	-	-	870	2 500
	Excrement odour	50	250	-	-	100	200	1 250	650	2 500
Initial concentration of components [µl/l]		30	50	10	5	4	8	3		

7.2.1 Preparation of the quasi sweat odour

7.2.1.1 Clean a plastic bag (6.1) by using the diluent gas (5.8).

7.2.1.2 Inject 1,7 l of the diluent gas into the plastic bag by using the air pump (6.2).

7.2.1.3 Inject 500 ml of isovaleric acid master gas, 250 ml of acetic acid master gas and 50 ml of ammonia master gas in order into the plastic bag by using the syringe (6.5) according to Table 3. See Figure 1.

Figure 1 — Injection of master gases

7.2.2 Preparation of quasi body odour (nonenal mixture odour)

7.2.2.1 Clean a plastic bag (6.1) by using the diluent gas (5.8).

7.2.2.2 Inject 870 ml of the diluent gas into the plastic bag by using the air pump (6.2).

7.2.2.3 Inject 830 ml of nonenal master gas, 500 ml of isovaleric acid master gas, 250 ml of acetic acid master gas and 50 ml of ammonia master gas in order into the plastic bag by using syringe (6.5) according to Table 3.

7.2.3 Preparation of quasi excrement odour

7.2.3.1 Clean a plastic bag (6.1) by using the diluent gas (5.8).

7.2.3.2 Inject 650 ml of the diluent gas into the plastic bag by using the air pump (5.2).

7.2.3.3 Inject 1 250 ml of indole master gas, 200 ml of methyl mercaptan master gas, 100 ml of hydrogen sulfide master gas, 250 ml of acetic acid master gas and 50 ml of ammonia master gas in order into the plastic bag by using syringe (6.5) according to Table 3.

8 Sensor response check

To confirm the response of the sensors, the measurements are performed with all odour chemicals by using the odour test instrument (6.9).

If a sensor shows no response, replace the sensor and check again.

9 Preparation of calibration curve

9.1 Initial concentration

The initial odour chemical concentrations of the test are shown in Table 3 for all component chemicals.

9.2 Odour unit concentration for quasi unpleasant odours

9.2.1 Odour unit concentration for the initial concentration

The odour unit concentration value is simply calculated by the initial concentration (b) divided by the olfactory threshold concentration (a) as shown in Table 4.

Table 4 — Odour unit concentration for the component chemicals

Unpleasant odour components	Ammonia	Acetic acid	Isovaleric aicid	Nonenal	Hydrogen sulfide	Methyl mercaptan	Indole
Olfactory threshold concentration [µl/l] (a)	1,5	0,006	0,000 078	0,000 08	0,000 41	0,000 07	0,000 3
Initial concentration of components [µl/l] (b)	30	50	10	5	4	8	3
Odour unit concentration (b)/(a)	20	8 333	128 205	62 500	9 756	114 286	10 000

9.2.2 Odour unit concentration for the quasi unpleasant odour

The largest odour unit concentration value of the component chemicals is selected as the odour unit concentration of the quasi unpleasant odour as shown in Table 5.

Table 5 — Odour unit concentration of the quasi unpleasant odours

Quasi unpleasant odour	Odour unit concentration for selection							
	quasi unpleasant odours	Ammonia	Acetic acid	Isovaleric acid	Nonenal	Hydrogen sulfide	Methyl mercaptan	Indole
Sweat odour	**128 205**	20	8 333	**128 205**				
Body odour (nonenal mixture odour)	**128 205**	20	8 333	**128 205**	62 500			
Excrement odour	**114 286**	20	8 333			9 756	**114 286**	10 000

9.3 Creation of calibration curve for quasi unpleasant odours

9.3.1 Preparation of dilution series

9.3.1.1 Clean 3 l of the plastic bags by using diluent gas.

9.3.1.2 Inject the master gas and diluent gas with the amount shown in Table 6 by using the air pump into the plastic bags.

Table 6 — Mixing recipe for dilution series

Dilution series	master gas (ml)	diluent gas (ml)	Total (ml)
Initial concentration	2 500	0	2 500
1/3	830	1 670	
1/10	250	2 250	
1/100	25	2 475	

9.3.2 Measurement

Obtain the response values for dilution series of all quasi unpleasant odours by using the odour test instrument.