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Textiles — Determination of deodorant property —

Part 1: General principle

Textiles — Détermination des propriétés de neutralisation d'odeurs —

Partie 1: Principe général

Reference number
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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

Unpleasant odours in daily human life are toilet odour, sweat odour, body odour (nonenal mixture odour), excrement odour, etc. Textile products that reduced these unpleasant odours from ambient air or around the human body were offered in the market by using advanced technology.

However, the evaluation method for such deodorant textiles did not develop as an International Standard. This fact has been making it difficult to evaluate correctly the deodorant property of the textile products for consumers as well as manufactures worldwide.

A current practical method for the evaluation of odour is a human sensory testing method in which a person is directly judged by the human sense of smell if there is an odour or not. This human sensory testing method is difficult to standardize as an objective indicator. Considering this situation, test methods using instruments or ultra-microanalysis testing methods have been developed.

Unpleasant odours are compounds of an infinitesimal quantity of chemicals. ISO 17299 provides a definition for the major component chemicals of odours and specifies the test methods by using several kinds of instruments in which the reduction rate of odour from ambient gas of the textile products is determined.

This part of ISO 17299 describes the general principle of testing methods for the deodorant property of textile products. Actual testing methods are described in ISO 17299-2 to ISO 17299-5.

Textiles — Determination of deodorant property —

Part 1: General principle

1 Scope

This part of ISO 17299 specifies the general principle of the deodorant textile products and deodorant testing methods for textile products, such as woven fabric, knit, nonwoven, fibres and yarns, braiding products, tapes and slings.

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*

3 Terms and definitions

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

3.1 General

3.1.1
unpleasant odour
uncomfortable odour related to the human living environment, such as toilet odour, sweat odour, body odour (nonenal mixture odour), and excrement odour, etc.

3.1.2
major component chemicals of unpleasant odour
chemicals that compose the unpleasant odour in the living environment

Note 1 to entry: These chemicals are as follows:

— toilet odour: ammonia;

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

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

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

Note 2 to entry: Other chemicals might be relevant. The selected chemicals are conceived as representative.

3.1.3
quasi-unpleasant odour
mixed odour produced artificially similar to unpleasant odour

Note 1 to entry: Mixed odours are produced artificially for the purpose of ISO 17299-5. The component chemicals for each odour are defined 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.

3.1.4
deodorant textile
textile with the ability to reduce the unpleasant odour in the ambient air around textile products

Note 1 to entry: The unpleasant odour is caused by the chemicals listed in 3.1.2 and 3.1.3.

3.1.5
deodorant property
ability to reduce the unpleasant odour in ambient air

Note 1 to entry: The unpleasant odour is caused by the chemicals listed in 3.1.2 and 3.1.3.

3.1.6
deodorant substance
substance able to interact physically or chemically with unpleasant odours

Note 1 to entry: The unpleasant odour are listed in 3.1.2 and 3.1.3.

Note 2 to entry: Examples of deodorant substances are shown in Annex A.

3.1.7
deodorant processing
process giving a deodorant property to textile products through a production process for application of deodorant substances on the surface or inside of the textile products by a padding and drying process, to be absorbed by a chemical treatment process, to be blended in the polymer by a spinning process, or to be applied using other techniques

3.2 Deodorant testing

3.2.1
evaluation of a deodorant property
measurement of the reduction of chemical concentration in a container with test specimen, comparing to without specimen

Note 1 to entry: The measuring methods of the concentration of odour chemicals are described in 3.2.2 to 3.2.6.

3.2.2
human sensory testing
judgement of strength of the smell by the human sense of smell

Note 1 to entry: The human sensory testing method is described in Annex C as an example. Although human sensory testing is not suitable for a standard, the procedure was used to develop the instrument testing method to determine the details of testing conditions.

3.2.3
detector tube method
concentration of odour component chemicals is measured by using the detector tube

Note 1 to entry: The detector tube method is described in ISO 17299-2 which is applied to the test of acetic acid, hydrogen sulphide, and methyl mercaptan.

3.2.4
gas chromatography method
concentration of odour component chemicals is measured by gas chromatography

Note 1 to entry: The gas chromatography (GC) method is described in ISO 17299-3, which is applied to the test of nonenal, indole, isovaleric acid, and the mixture liquid of acetic acid and NaCl.

3.2.5
condensation sampling analysis
odour component chemicals are condensed by the condensation sampling method for each chemical, and then, the appropriate analytical instruments are chosen to obtain the concentration of odour chemicals

Note 1 to entry: The method is described in ISO 17299-4 for the test of indole, methyl mercaptan, hydrogen sulfide, isovaleric acid, and nonenal.

3.2.6
metal-oxide semiconductor sensor method
concentration of quasi-unpleasant odour chemicals are measured by using the metal-oxide semiconductor sensors

Note 1 to entry: This method is described in ISO 17299-5 for the test of quasi-sweat, quasi-body odour (nonenal mixture odour), and quasi-excrement odour.

4 Principle

Concentration of gaseous odour component chemicals of the gas in containers with a specimen denoted as A, and without specimen denoted as B, is measured by using the specified instruments after the designated contacting time. The reduction rate is determined from the chemical concentration with and without specimen as $(B - A)/B \times 100$ %. The test shall be done either with the odour chemical individually or with mixed odour component chemicals as described in each part of ISO 17299.

5 Reagents

Unless otherwise specified, analytical grades have to be used.

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

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

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

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

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

5.6 **Isovaleric acid**, solution with purity of 98,0 %.

5.7 **2-Nonenal ($C_9H_{16}O$)**, reagent with purity of 95,0 %.

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

NOTE Nitrogen gas obtained from a nitrogen cylinder (purity of at least 99,999 9 %) can be used.

5.9 **Ethanol**, reagent with purity of 99,5 % in water.

6 Materials and apparatus

6.1 **Plastic bag**, made of vinyl fluoride film, polyester and polyester laminated film, polyvinyl alcohol film, etc. A volume of 1 l, 3 l, 5 l, and 50 l is available depending on the purpose. A plastic or rubber tube is installed to the bag before testing.

6.2 **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.

6.3 **Integrating flow meter**, capable of measuring the gas flow of 500 ml/min or more.

6.4 Hand dryer, with an electric consumption of 1 kWh to be used to warm up ammonia to 40 °C to 50 °C to evaporate in the 1 l bag.

6.5 Syringe, made of a glass cylinder with a capacity of 0,5 ml, 100 ml, and 200 ml.

6.6 Micro-syringe, with a capacity of 10 µl.

6.7 Heat seal, to seal the opened parts of the plastic bag. The tape can be used with the same sealing capability as an alternative.

6.8 Aspirator, capable of evacuating all air in the 5 l plastic bag after sealing. A vacuum pump could be used.

6.9 Airtight stopper.

6.10 Oven, capable of using at 80 °C

6.11 Parmeator or cylinder, alternative standard gas generator.

7 Testing environment and sample conditioning

The testing environment shall be kept at a temperature of 20 °C and relative humidity of 65 % in accordance with ISO 139. The samples are conditioned under the same condition for at least 24 h.

NOTE The condition with a temperature of 23 °C and relative humidity of 50 % according to ISO 139 can be used for this test, then record the condition used on the test report.

8 Test procedure

The test procedure will be described in ISO 17299-2 to ISO 17299-5 as follows:

— ISO 17299-2*: Detector tube method*;

— ISO 17299-3*: Gas chromatography method;*

— ISO 17299-4*: Condensation sampling analysis*;

— ISO 17299-5*: Metal-oxide semiconductor sensors method*.

In all parts, the concentration of testing gas without a specimen and with a specimen are denoted as B and A, respectively.

9 Odour reduction rate

The reduction rate of the concentration is calculated from Formula (1).

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

where

ORR is the odour reduction rate in percentage;

B is the average of the concentration of testing gas without a specimen;

A is the average of the concentration of testing gas with a specimen.

10 Determination of deodorant property of the textile products

The purpose of this test method is to determine the deodorant property of textile products.

The labelling for the appropriate deodorant property should be presented when the chemical concentration reduction is larger than the values referred to in Annex E as an example.

11 Test report

The following items are at least recorded in the test report:

a) a reference to this part of ISO 17299 (i.e. ISO 17299-1) and the test method used;

b) kind, origin, and designation of the sample (partial specimen, if applicable);

c) individual odour concentration data, average, and reduction rate;

d) any deviation from this part of ISO 17299.

Annex A
(informative)

Deodorant substances

A.1 Deodorant substances

Table A.1 shows substances which are supposed to have anti-bacterial property as well as deodorant property.

Table A.1 — Deodorant substances

Category I	Category II	Deodorant substances
1) Inorganic	[Metallic salt]	Molecular sieves, crystalline aluminosilicate silver, or sodium (silver displacement zeolite)
		Silver/zinc zeolite
		Silver/zeolite
		Zirconium phosphate/silver oxide
		Zirconium phosphate, silver oxide/zinc oxide
		Titanium phosphate, zinc oxide, and titanium oxide gel compound
		Compounds of titanium phosphate silver supported gel and zinc oxide
		Silver supported dioxide silicate
		Silver oxide, triphosphoric acid ammonium, and sodium phosphate compound
		Silver chloride
		Silver
		Zinc oxide
		Copper
		Copper compound
		Tetra-amine copper ion
		Glass of phosphoric acid system
		Hydrophilic amino silicon polymer including metallic oxide
	[Carbon system]	Steam-activated carbon
2) Organic	[Biguanide]	Chlorohexidine gluconate
		Chlorohexidine gluconate and poroctone olamine
		Polyhexamethylene biguanide hydrochloride
		Copolymer of chlorohexidine and 2-propenamide 2-methyl propane sulfonic acid
		Composition of polyhexamethylene biguanide and zinc oxide
	[Carbanilide]	Triclocarban
		Composition of triclocarban and nalidixic acid
		Phenyl amide compound

Table A.1 *(continued)*

Category I	Category II	Deodorant substances
2) Organic	[Ampholytic surfactant]	Alkyl amide propyl dimethyl β-hydroxyethyl ammonium salt and poly [oxyethylene (dimethylamino) ethylene (dimethylamino) ethylene chloride]
	[Carboxylic acid]	Polymethacrylic acid
		Compounds of polyacrylate and zinc sulfate
		Nalidixic acid: 1 ethyl-1, 4-dihydro-7-methyl-4-oxo-1, 8-naphthyridine-3-carboxylic acid
	[Alcohol]	Polyhydric alcohol system compound
	[Quaternary ammonium salt]	Benzalkonium chloride
		Organic silicone quaternary ammonium salt
		N-polyoxyalkylene-N, NN-tri-alkylene-ammonium salt
		Alkyl quaternary ammonium and carboxylate salt
		Alkyl dimethyl ammonium salt
		Alkyl dimethyl benzal conium salt
		Alkyl quaternary ammonium salt
		N, N, NN-tetra alkyl quaternary ammonium salt
		Cetyl tri-methyl ammonium chloride
		Di-alkyl quaternary ammonium salt
		Tetra-alkyl quaternary ammonium salt
		Octa-decyl di-methyl ammonium chloride
		Ammonium chloride di-decyl di-methyl
		Di-decyl di-methyl ammonium chloride
		Compounds of quaternary ammonium chloridization of copolymer of phosphoric acid ester monomer
		3-(methoxysilyl)-propyl octadecyl dimethyl ammonium chloride
		N-polyoxyalkylene-NN,-tri-alkylene-ammonium
		Benzalkonium chloride and polyhydric alcohol system compound
		Alkyl trimethyl ammonium dibutyl phosphate acid salt
		Dicyanamide and diethylene triamine and ammonium chloride condensate
		Dicyandiamide poly-alkylene-poly-amine-ammonium polycondensation body
		Cation polymer
		Partial escape acetyl compound of (poly-β-1,4) N-acetyl-D glucosamine and reaction product of hexamethylenebis (3-chloro –2- hydroxypropyl dimethyl ammonium chloride)
	[Phenol]	Alky-lenebis phenol sodium salt
		Para-chrol-metaxylenol
		Bis(2,6 di-t-ptyl-4-methylic phenol) pentaerythritol diphosphate
	[Amino acid]	N-alkyloyl-L -glutamine acid silver copper
	[Sulfamide]	N, N-dimethyl-N'-fluoro-dichloro-methylthio-N"-phenylsulf-amide
	[Pyridine]	Bis(2-pyridinethiol-zinc-1-oxide)zinc Bis[1- hydroshiki-2(1) pyridiothionate (0,S)-T-4] zinc

Table A.1 *(continued)*

Category I	Category II	Deodorant substances
2) Organic	[Nitrile]	2, 4, 5, 6 tetra-chloro-isophthalonitrile
	[Polymer]	Acrylonitrile and acrylate copolymerization copper cross-linked
		Acrylonitrile sulfuration copper complex
		Acrylamide-diallylamine hydrochloride copolymer
		Methacrylate copolymerization
	[Others]	Flowable sulfur
		Lactoferin and lactoferishin
3) Natural organic	[Glucidic]	Chitosan
		Hydroxy propyl chitosan
		Chitosan of cross-bridge
		Chitosan organic acid salt
		Chitosan fine powder (polyglucosamine)
		Chitin fibre
		Chitin
	[Tropolone]	Japanese cypress thiol
		Japanese cypress oil emulsion
		Leaf oil
		Compound of cyclodextrins and leaf oil
		Leaf oil emulsion
	[Ester]	Undecylenic acid monoglyceride
		Fatty acid glyceride
		Fatty acid ester acid (propylene glycol monoester)
		Glycerine fatty acid ester
		Methoxypolyethyleneglycolmethacrylate-alkyl phosphoric acid ester
		Phosphoric acid ester system polymer
	[Terpene]	1,8-cineole (felon herb, eucalyptus, and lemon eucalyptus)
4) Others		Copper Included cellulose textile

Annex B
(informative)

Odour components and deodorant

B.1 General

The major component chemicals for odours are given as Table B.1.

Table B.1 — Major component chemicals for odours

Odour component chemicals		Kind of odour				
		Sweat	Body	Excrement	Cigarette	Garbage
Ammonia	NH_3	m	m	m	m	m
Acetic acid	CH_3COOH	m	m	m	m	
Isovaleric acid	$CH_3(CH_2)_3COOH$	m	m			
Nonenal	$C_9H_{16}O$		m			
Acetaldehyde	C_2H_4O				m	
Methyl-mercaptan	CH_4S			m		m
Hydrogen sulfide	H_2S			m	m	m
Indore	C_8H_7N			m		
Pyridine	C_5H_5N				m	
Tri-methylamine	C_3H_9N					m
m Major component.						

B.1.1 Sweat odour

Approximately 99 % of sweat is water, but some fatty acid esters of glycerol come out together from glandulae sudoriferae and stain textiles, then the fatty acid esters are biologically degraded and through a chemical reaction path, acetic acid, ammonia, etc. are formed. Even acetic acid never comes out from the human body directly as sweat.

The selection of chemicals for the test is based on the information above.

B.1.2 Body odour (nonenal mixture odour)

Nonenal was found as a component of body odour (nonenal mixture odour) in 2001.

B.2 Deodorant mechanism and substances

The deodorant mechanism and used substances are summarized in Table B.2.

Table B.2 — Summary of deodorant mechanism and used substances

Deodorant mechanism		Details	Used substances
Chemical reaction	Oxidation-reduction reaction Addition/condensation reaction	Chemicals are resolved with the oxidant and reducing agent. Odour chemicals are removed by addition and the condensation reaction.	— Hydrogen peroxide — Sodium sulfite — Photocatalytic TiO_2 — Glyoxal — Methacrylate ester
	Neutralization reaction Ion-exchange reaction	Acidic and basic odour are removed by the neutralization reaction.	— Multivalent carboxylic acid — Multivalent phenol
Physical absorption	Physical absorption	Odour chemicals are absorbed to the surface of porous materials.	— Activated carbon — Zeolite
Sensory deodorant	Masking action	Odours are covered by strong smells.	— Flavor and fragrance — Tree acetic acid
	Offset action	Odours are offset by other odorous chemicals.	— Turpentine — Eucalyptus
Biological deodorant	Enzyme action	Organic odorous chemicals are decomposed by oxidation or microorganism.	— Hydrolase — Yeast

Annex C
(informative)

Human sensory testing method

C.1 General

The human sensory testing method for odour has been used for a long time. This is a method using the human sense of smell as a detector.

However, the testing procedure and the finding from the human sensory testing method could be reflected in a standard using the instrumental devices to test odour.

Therefore, the testing procedure of the human sensory testing method is explained in Annex C for information.

C.2 Level of odour strength

Designation of the odour strength level into six stages as described below.

— 0: odourless;

— 1: faint odour without knowledge of the kind of odours (odour threshold concentration);

— 2: weak odour with knowledge of the kind of odours (conceivable threshold concentration);

— 3: medium odour, can be easily perceived;

— 4: strong odour;

— 5: very strong odour.

C.3 Testing method

C.3.1 Testing environment

The testing environment should be kept at a temperature of 20 °C and relative humidity of 65 %, or 23 °C and relative humidity of 50 %, in accordance with ISO 139. The condition used should be described in the test report.

C.3.2 Container

Conical flask, 500 ml

C.3.3 Object-odour component chemicals

Ammonia, acetic acid, isovaleric acid, nonenal, hydrogen sulfide, indole, and methyl mercaptan.

Warning — Hydrogen sulfide is a very toxic chemical and is harmful to humans. The test should be performed under strict control and by a trained expert with sufficient knowledge.

C.3.4 Panellist

Six persons or more who should be trained to recognize and quantify the odorant molecules. The odour threshold should be measured for all panelists before testing and the result should be included in the report.

C.3.5 Odour concentration testing procedure

An original odour gas is diluted to 30 times, 100 times, 300 times, 1 000 times, and 3 000 times, for example, and the odour gas is inserted into a conical flask and is tested by panelists.

NOTE The original odour gas concentration of the chemical is detected by an instrument and the concentration of the diluted gas is obtained by calculation, based on the dilution magnification.

C.4 Result

The odour chemical concentration for the odour strength, 3,5 and 2,0 of each odour component chemical, was determined as shown in Table C.1 from the human sensory testing.

Table C.1 — Concentration of the odour component chemical corresponding to human sensory test, relating to odour strength

Odour element chemicals	Odour strength 3,5 concentration µl/l	Dilution ratio to make odour strength 2,0
Ammonia (NH_3)	105	1/10
Acetic acid (CH_3COOH)	1 to 4	1/10
Isovaleric acid $(CH_3)_2CHCH_2COOH$	0,1	1/20
Hydrogen sulfide (H_2S)	0,2	1/5
Methyl mercaptan(CH_3SH)	1	1/30
Indole (C_8H_7N)	1,0	1/50
Nonenal ($C_6H_{13}CH = CHCHO$)	0,03	1/20

Annex D
(informative)

Determination of the testing condition of the instrument testing method

D.1 General

The initial concentration of the odour component chemical for the instrument testing method is considered for evaluating the detector tube method and the gas chromatography method, because the odour concentration for strong/medium odour 3,5 is under µl/l level for some odour chemicals which makes testing difficult.

However, the condensation sampling analysis can be used for the initial concentration directly from the human sensory testing of 3,5 level, because of very high sensitivity.

D.2 Determination of initial concentration for detector tube method (ISO 17299-2) and gas chromatography method (method A of ISO 17299-3)

The initial concentration for the instrument devices was evaluated at several points of concentration and finally determined as in Table D.1, considering the availability of the detector tube and the reproducibility.

The deodorant textile samples are prepared as samples with an odour strength of less than 2,0 by human sensory testing and with non-deodorant treatment control.

The samples were tested using several initial concentrations and finally determined the values shown in Table D.1. The testing results were obtained with a good separation.

Table D.1 — Initial concentration of odour chemicals for testing method

Odour component chemicals	Initial concentration of chemical µl/l	Testing method
Ammonia	100	Part 2 Detector tube
Acetic acid	30	Part 2 Detector tube
Isovaleric acid	38	Part 3 A Gas chromatography
Hydrogen sulfide	4	Part 2 Detector tube
Methyl mercaptan	8	Part 2 Detector tube
Indole	33	Part 3 A Gas chromatography
Nonenal	14	Part 3 A Gas chromatography

D.3 Condensation sampling analysis (ISO 17299-4)

The initial odour chemical concentration was selected, referring to the human sensory testing method shown in Table C.1.

D.4 Metal-oxide semiconductor method

The preparation of testing gas is described in (ISO 17299-5). The initial concentration is chosen as a good response of these metal-oxide semiconductor sensors as the nearest values compared with other methods.

Table D.2 — Initial concentration of odour chemicals for metal-oxide semiconductor method

Odour component chemicals	Initial concentration of chemical µl/l
Ammonia	30
Acetic acid	50
Isovaleric acid	10
Hydrogen sulfide	4
Methyl mercaptan	8
Indole	3
Nonenal	5

Annex E
(informative)

Deodorant textile certification (a practical reference)

E.1 Deodorant textile certification

One certification system for deodorant textiles has been organized in the market in Japan. The major configuration of the certification system is described below.

E.2 Certification requirement

E.2.1 Labelling

— certification organization identity;

— certification number;

— deodorant treatment for: toilet, ammonia, sweat odour, body odour (nonenal mixture odour), excrement odour;

— deodorant effect expression: (This textile product reduces the unpleasant odour from the ambient air due to direct contact with the odour component chemicals.);

— odour component chemicals: refer to 3.1.2;

— deodorant substances: to identify the substances used for deodorant treatment;

— name of manufacture.

E.2.2 Safety requirement of deodorant substances and products

The deodorant substances and products should be complied to the existing domestic laws and to the safety rule of the certification standard, such as to execute a skin allergic test, patch test, for the textile products.

E.2.3 Deodorant property requirement

The significant deodorant effect is determined by the minimum concentration reduction rate for each chemical as shown in Table E.1. From long experience, when the reduction rate is equal to or more than the value given in Table E.1, the deodorant effect could be perceptible in the situation.

Table E.1 — Criteria of the deodorant textiles for each odour component chemical

Odour component chemical	Minimum % of concentration reduction rate
Ammonia	70
Acetic acid	70
Isovaleric acid	85
Hydrogen sulfide	70
Methyl mercaptan	70
Indole	70
Nonenal	75

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[6] ISO 17299-4, *Textiles — Determination of deodorant property — Part 4: Condensation sampling analysis*

[7] ISO 17299-5, *Textiles — Determination of deodorant property — Part 5: Metal-oxide semiconductor sensors method*.

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