INTERNATIONAL STANDARD

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Textiles — Determination of deodorant property —

Part 4: Condensation sampling analysis

Textiles — Détermination des propriétés du déodorant —

Partie 4: Analyse par condensation d'échantillonnage

Reference number
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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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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).

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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 of *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 condensation sampling analyses to measure the very low concentration of the odour chemicals which is assumed at the level of human olfactory perception threshold.

Textiles — Determination of deodorant property —

Part 4:
Condensation sampling analysis

1 Scope

This part of ISO 17299 specifies a deodorant testing method using analytical instruments after using condensation sampling methods for all textile products. This method is applicable to gaseous odour chemicals, such as indole, methyl mercaptan, hydrogen sulfide, isovaleric acid, and nonenal.

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

3 Terms and definitions

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

3.1
condensation sampling
method for collecting gaseous odour chemicals at very low concentration levels, which are then condensed by analytical methods to higher concentration levels to facilitate detections of the chemicals

4 Principle

The concentration of a gaseous odour chemical in a container with or without a textile specimen after a designated elapsed time is measured by instrumental devices. The reduction rate of concentration of gaseous odour chemicals is calculated from the difference of average concentration data obtained from the container with specimen and from that without specimen. The test shall be done for each chemical individually.

5 Reagents

Unless otherwise specified, analytical grade reagents are used.

5.1 Ethanol, reagent with concentration of 99,5 %.

5.2 Odour chemicals.

5.2.1 Indole (C_8H_7N), reagent.

5.2.2 Methyl mercaptan, at a certified concentration of 2,0 µl/l ± 3 % in nitrogen diluent.

5.2.3 Hydrogen sulfide (H_2S), at a certified concentration of 2,0 µl/l ± 3 % in nitrogen diluent.

5.2.4 Isovaleric acid [$(CH_3)_2CHCH_2COOH$], reagent with a purity of 98,0 %.

5.2.5 Nonenal ($C_9H_{16}O$), reagent with a concentration of 95,0 % in ethanol solution.

5.3 Diluent gas, nitrogen gas from nitrogen gas cylinder with a purity of at least 99,99 %.

6 Apparatus and materials

6.1 Plastic bags, with a volume of 3 l, at least six plastic bags for one test (see ISO 17299-1).

6.2 Micro syringe, capable of delivering 10 µl of solution by an accuracy of ±0,1 µl.

6.3 Heat sealer, used to seal open edge of plastic bag. The adhesive tape can be used as an alternative.

NOTE A sealing attachment such as a linear binder can be used.

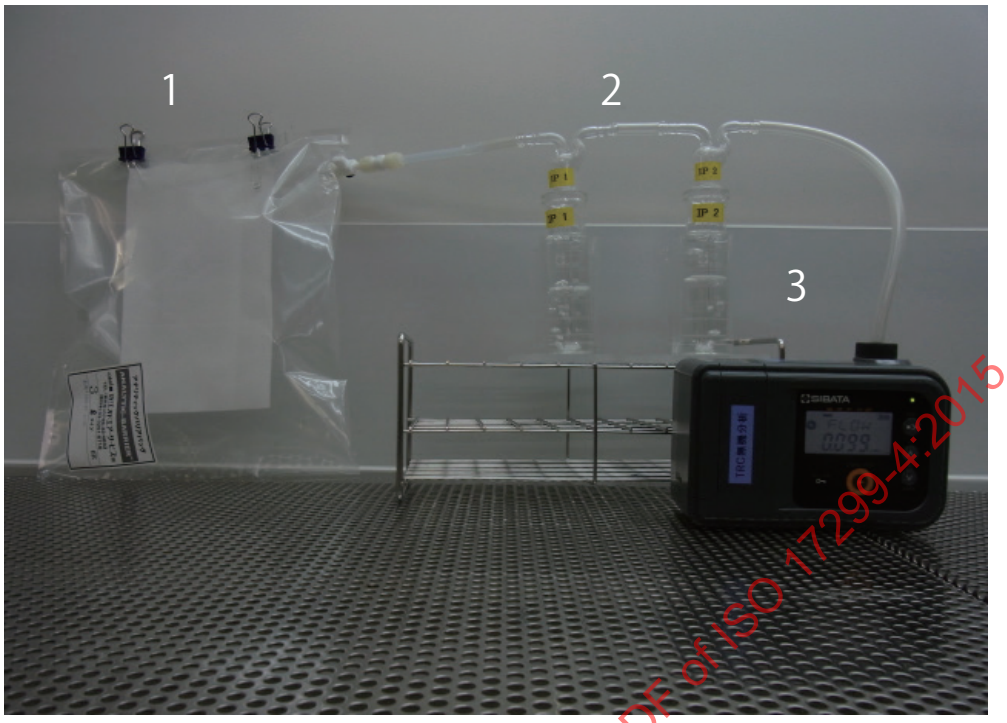
6.4 Aspirator or vacuum pump, capable of evacuating remaining air from 3 l sealed plastic bag (refer to Figure 1, item 1).

6.5 Adsorption tube, capable of being mounted on a thermal desorption instrument, and absolving condensed samples of testing chemicals.

6.6 Thermal desorption instrument, capable of mounting an adsorption tube and baking it for cleaning at a temperature over 280 °C with flow of inert gas.

6.7 Impinger, capable of putting absorbing solution and condensing odour chemical of the gas sample supplied from the plastic bag (refer to Figure 1, item 2).

6.8 Air pump, capable of drawing approximately 0,2 l per minute of a sample gas from a plastic bag into adsorption tube, impinger, etc. (refer to Figure 1, item 3).



Key

1 plastic bag with specimen
2 impinger
3 air pump

Figure 1 — Sampling set-up

6.9 Thermal desorption gas chromatography (GC) equipment, coupled with a thermal desorption instrument which can be heated up a temperature higher than 280 °C and equipped with a flame photometer detector (FPD), a flame ionization detector (FID), or mass spectrometer (MS).

6.10 Ion chromatography (IC) equipment, to analyse the testing solution of condensed sample in impinger.

The detectors used are Conductivity Detector (CD).

7 Test method

7.1 Sampling of test specimen

The dimension or mass of specimen for textile products is shown in Table 1.

Table 1 — Dimension or mass of specimen

Kind	Dimension or mass
Fabrics (woven, knit, non-woven, and tapes)	250 cm^2 ± 12,5 cm^2
Yarns, fibres, and feather	2,5 g ± 0,125 g

For multi-layer products, the edge and the untreated layer (or the "not targeted" layer) may be covered with an aluminium sheet or the test specimen may be folded in two with the untreated layers inward, in order to avoid contact of untreated layer with odorous atmosphere.

Condition the specimen as specified in ISO 17299-1.

7.2 Preparation of container

7.2.1 Prepare six plastic bags with the capacity of 3 l, three plastic bags for testing with a specimen and three plastic bags for the control test without a specimen. Place adhesive tape on the plastic bag at the insertion point of the odour chemical.

NOTE The capacity of plastic bag is 3 l for all testing methods.

7.2.2 Clean the plastic bags used in this test using nitrogen gas or clean air, more than five times of taking gas in and out by filling gas of the plastic bag volume just before testing.

7.3 Preparation of odour chemical solution or master gas

Perform the test for each odour chemical independently. Prepare the odour chemical solutions for the testing as indicated below.

7.3.1 Indole solution.

Dissolve 0,6 g of the indole reagent (5.2.1) in 100 ml of ethanol (5.1).

7.3.2 Methyl mercaptan

Prepare the master gas with 1 µl/l of methyl mercaptan concentration by using the standard methyl mercaptan gas (5.2.2) and diluent gas (5.3).

7.3.3 Hydrogen sulfide

Prepare the master gas with 0,2 µl/l of hydrogen sulfide concentration by using the standard hydrogen sulfide gas (5.2.3) and diluent gas (5.3).

7.3.4 Isovaleric acid.

7.3.4.1 Dilute isovaleric acid reagent (5.2.4) of 5 ml and make up to 100 ml by ethanol (5.1).

7.3.4.2 Dilute the diluted solution of 1 ml and make up to 100 ml by ethanol (5.1).

7.3.5 Nonenal.

7.3.5.1 Dilute nonenal reagent (5.2.5) of 3,5 ml and make up to 100 ml by ethanol (5.1).

7.3.5.2 Dilute the diluted solution of 0,6 ml and make up to 100 ml by ethanol (5.1).

Perform the test for each odour chemical separately.

7.4 Testing procedure

This is a test with specimens.

7.4.1 Take three specimens from the sample for one test as shown in Table 1.

7.4.2 Place the specimens in the plastic bag one by one, so as to spread them out as much as possible. See Figure 2.

NOTE Curling and creasing of specimen might be unavoidable.

Figure 2 — Specimen in a plastic bag

7.4.3 Hold the specimen by clips from outside of bag to make easier operation. Then, seal the opened edge of the bag for insertion of specimen by using heat sealer (6.3).

7.4.4 Deaerate from the plastic bag as much as possible by using an aspirator or a vacuum pump (6.4).

7.4.5 Insert 2,5 l of nitrogen gas or the odour testing gas prepared. Then, add the odour testing solution by using micro syringe into the dilution nitrogen gas. The injection needle is stung at the point of the attached adhesive tape. The insertion quantity is indicated in Table 2.

7.4.6 When inserting the solution or gas, be careful not to touch the specimen. After inserting the chemical, close the hole of the needle with adhesive tape.

Table 2 — Insertion quantity of odour testing solution or gas

	Indole	Methyl mercaptan	Hydrogen sulfide	Isovaleric acid	Nonenal
Odour testing solution	2,5 µl	—	—	2,5 µl	2,5 µl
Odour testing gas	-	2,5 l (1 µl/l)	2,5 l (0,2 µl/l)	—	—

7.4.7 Knead the bag 20 times.

7.4.8 Place the plastic bag still for 2 h as a reaction time.

7.4.9 After 2 h have elapsed, knead the bag 20 times.

7.4.10 Connect the outlet of the testing bag to either

— a plug of adsorption tube, or

— an impinger.

7.4.11 Extract 2 l of the testing gas from the plastic bag using the air pump (6.8) at a rate of 0,2 l/min and deliver the testing gas into a condensation sampling device as shown in Table 3.

Lie the testing bag sprawled during delivery of the testing gas.

Table 3 — Testing gas sampling method and amount

	Indole	Methyl mercaptan	Hydrogen sulfide	Isovaleric acid	Nonenal
Gas condensation sampling method	adsorption tube	impinger	impinger	adsorption tube	adsorption tube
Testing gas amount	2,0 l				

If using the activated carbon type adsorption tube, clean the tube by baking prior to testing.

7.5 Measurement of odour gas concentration

7.5.1 General

Mount an adsorption tube after sampling of the odour chemical on a thermal-desorption GC, then measure the gas concentration of the odour chemical. FID, FPD, or MS are available as the detector of GC.

If using methyl mercaptan and hydrogen sulfide, the absorbing solution after sampling by impinger could be measured by an ion chromatography.

7.6 Testing without specimen

This is a control test which is performed following the instructions in 7.4 and 7.5 without a specimen. The number of tests shall be same as that for the testing with specimen, i.e. three.

8 Calibration

Prepare three different concentration gases from the standard solution or master gas.

Measure the standard solutions or gases using either thermal-desorption GC or ion chromatography. Then, plot the peak area data on a graph against the inserted quantity of the odour chemical which is calculated from the concentration. More than three points of concentration are required and the coefficient of correlation shall be higher than 0,99.

9 Test result

9.1 Calculation of the concentration of the odour chemical

The peak area value from a gas chromatography is obtained for testing with or without the specimen.

The absolute amount (ng) of the odour chemical is obtained from the coefficient of the calibration curve, and the concentration of the odour chemical (ng/l) is calculated as follows:

The concentration of the odour chemical (ng/l) is equal to the odour component chemical amount (ng) divided by the sampling gas amount (l).

9.2 Calculation of reduction rate for odour chemical

The odour reduction rate (ORR) % is calculated by the following formula based on the calculated concentration in 9.1.

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

where

ORR is the odour reduction rate, in percentage;

A is the average of the concentration of chemical testing with specimen;

B is the average of the concentration of chemical testing without specimen.

10 Test report

The following items are at least recorded in the test result.

a) reference to this part of ISO 17299;

b) any useful information for the samples such as kind, origin, and designation of the sample (partial specimen, if applicable);

c) sampling procedure;

d) testing condition used;

e) individual chemical concentration data, average, and reduction rate;

f) any deviation from this part of ISO 17299.

Annex A
(informative)

Actual application of the thermal desorption GC-MS to the measurement of indole, isovaleric acid, and nonenal

A.1 General

The analytical instruments have the wide variety in the kind of type or specification among the laboratories, so the measurement conditions are unique to each instrument, even for the measurement of the same odour chemicals. To make a convenience for users, the example of typical set of measurement condition and the results are shown below as a reference.

A.2 Analytical conditions for the thermal desorption GC-MS

Primary thermal desorption	20 °C to 60 °C per minute to 280 °C (10 min hold)
Temperature of transfer line	300 °C
Secondary adsorption temperature	– 40 °C (used the insert of Tenax TA®[a]
Secondary thermal desorption	– 40 °C to 12 °C per second, to 300 °C (5 min hold)
Column	DB-5MS 30 m × 0,25 mm ID thickness of film 0,25 µm
Column temperature	40 °C to 300 °C, 10 °C per minute heating
Carrier gas	He, 1,0 ml per minute
Split ratio	50:1
Ionization	Electronic ionization (EI)
Ionization temperature	230 °C
Detector mode	Selected Ion Monitoring (SIM)
Monitor ion (*m/z*)	117 for indole to the quantitative analysis, 90 for indole to identify

[a] Tenax TA® 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.

See Figure A.1 to Figure A.9.

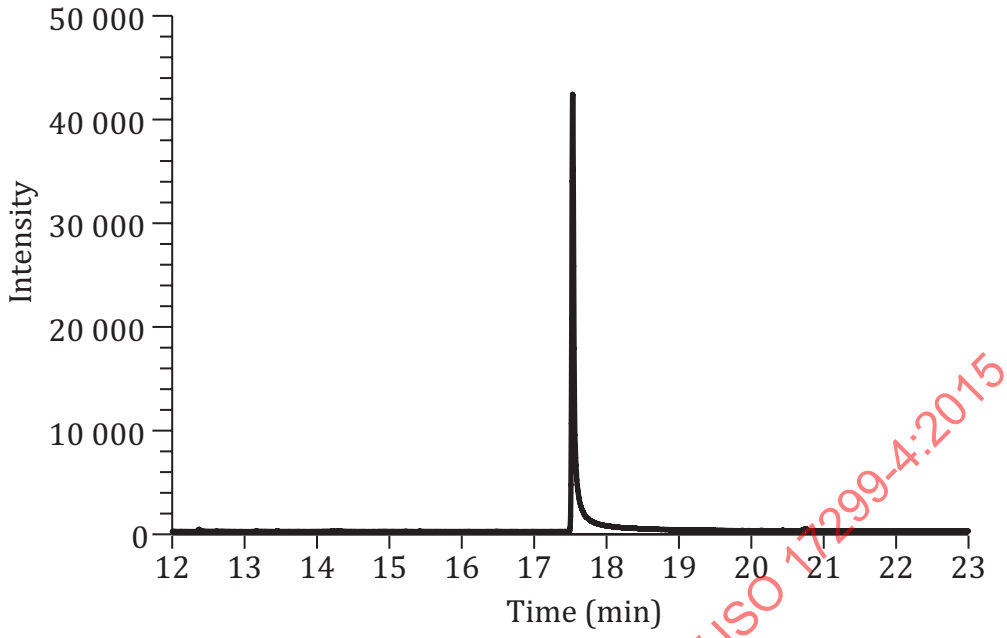


Figure A.1 — GC-MS-SIM Chromatogram of indole standard (21 ng)

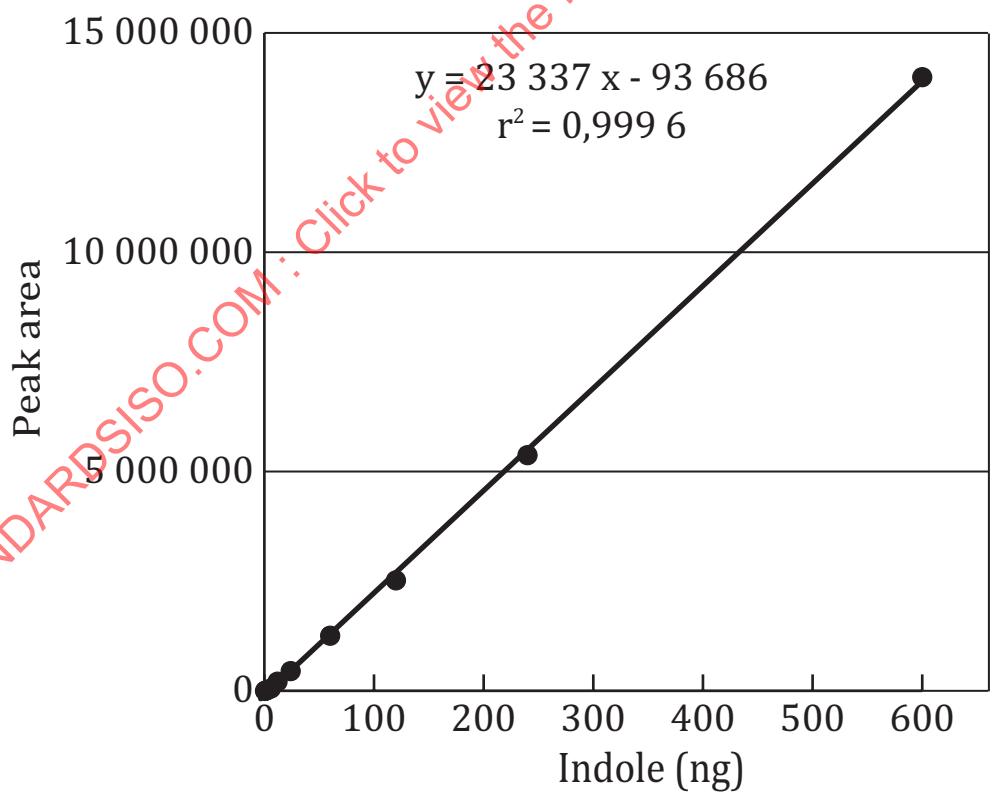


Figure A.2 — Calibration curve for Indole (6 ng~600 ng)

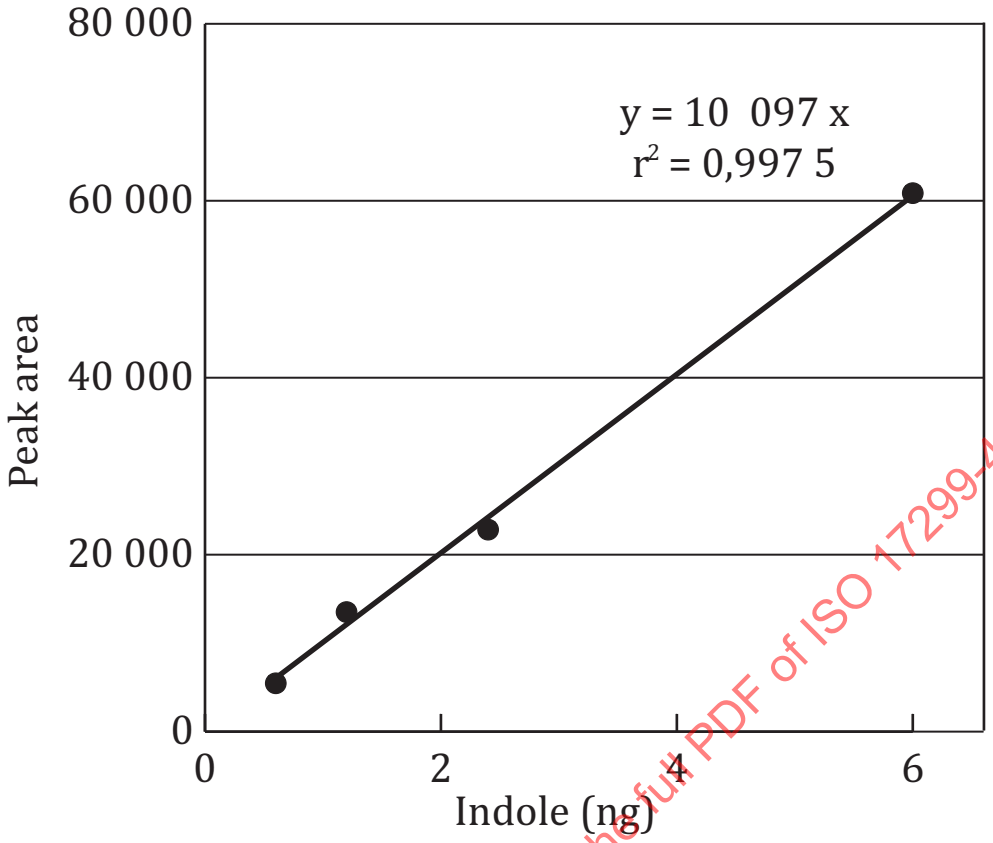


Figure A.3 — Calibration curve for indole (0,6 ng~6 ng)

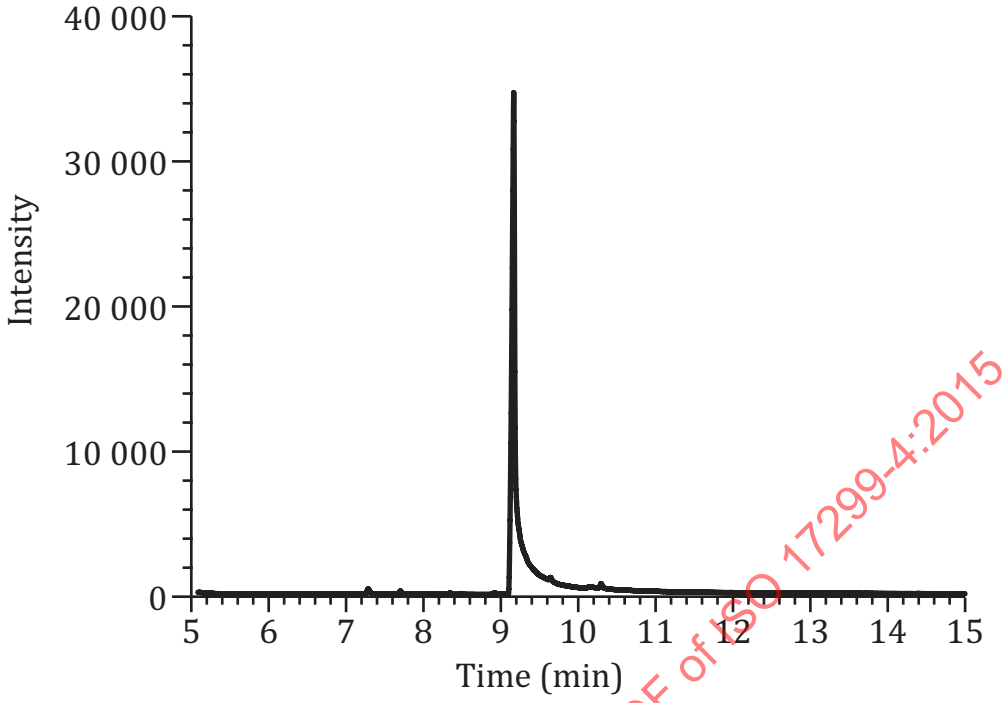


Figure A.4 — GC-MS-SIM chromatogram of isovaleric acid standard (55 ng)

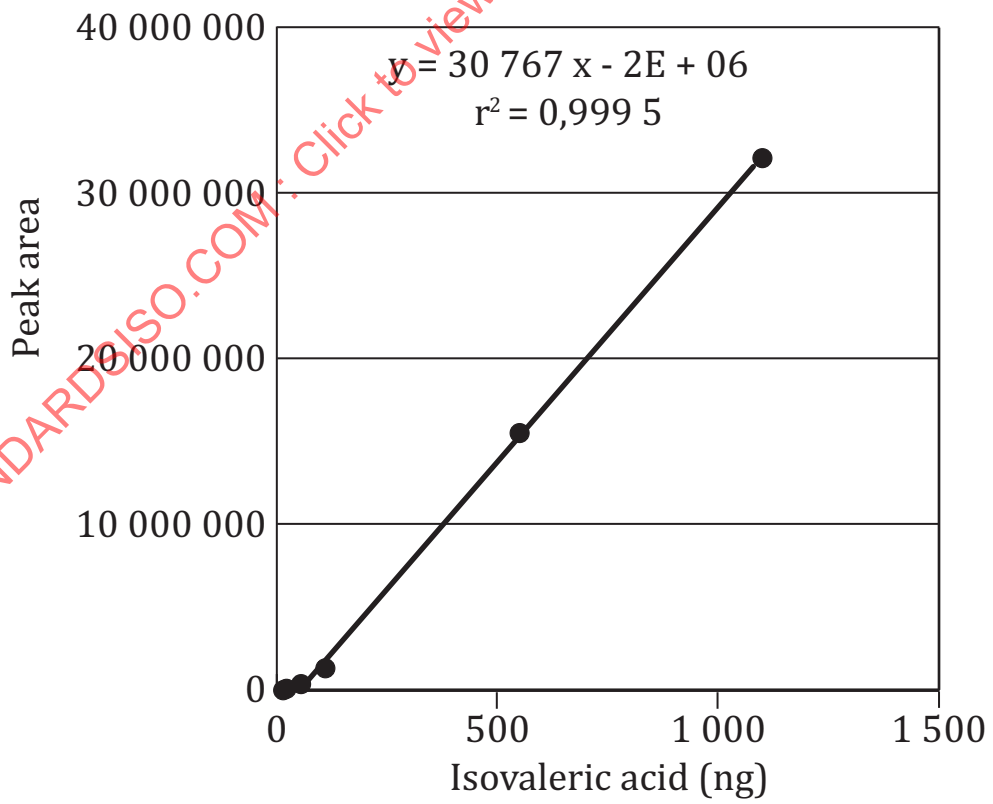


Figure A.5 — Calibration curve for isovaleric acid (55 ng~1 100 ng)

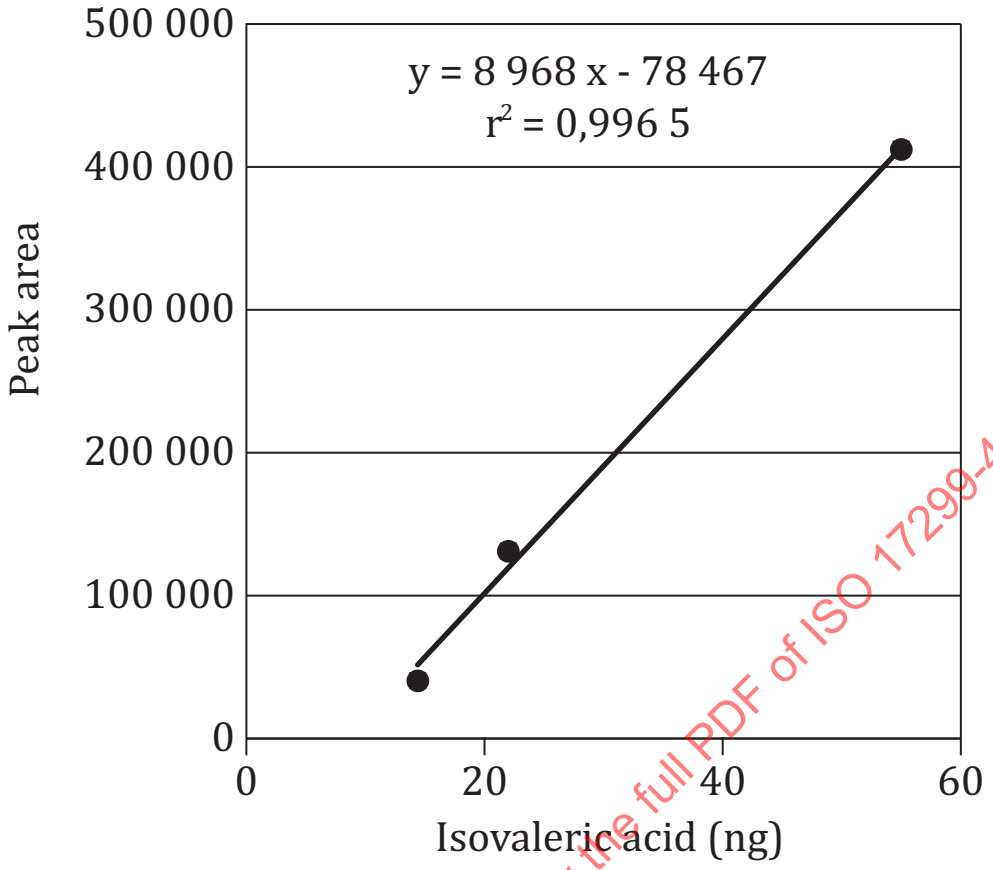


Figure A.6 — Calibration curve for isovaleric acid (14 ng~55 ng)

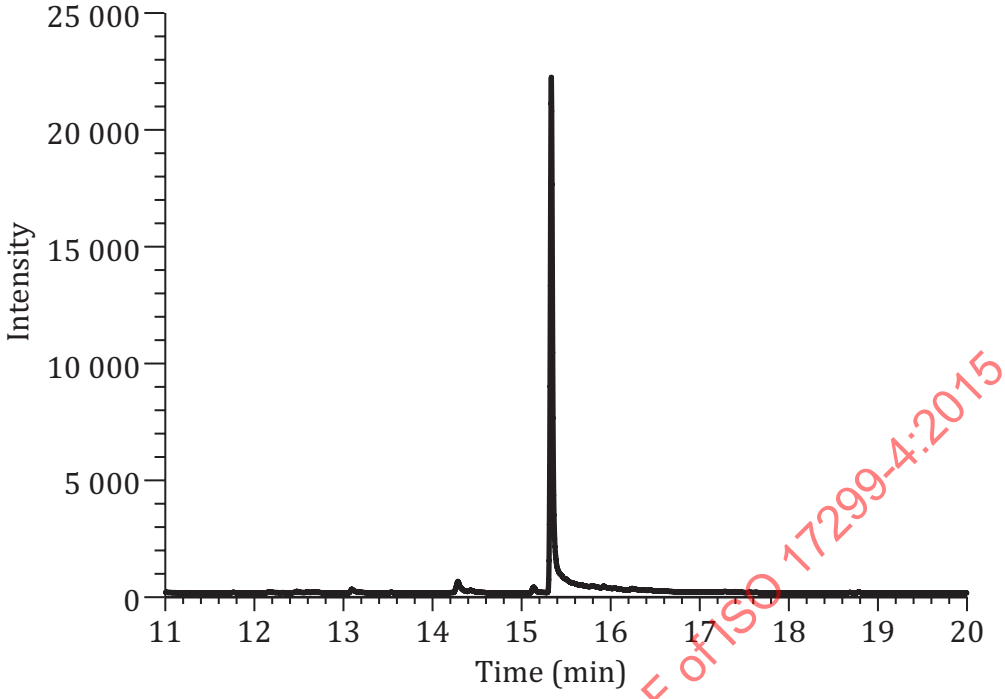


Figure A.7 — GC-MS-SIM chromatogram of nonenal standard (47 ng)

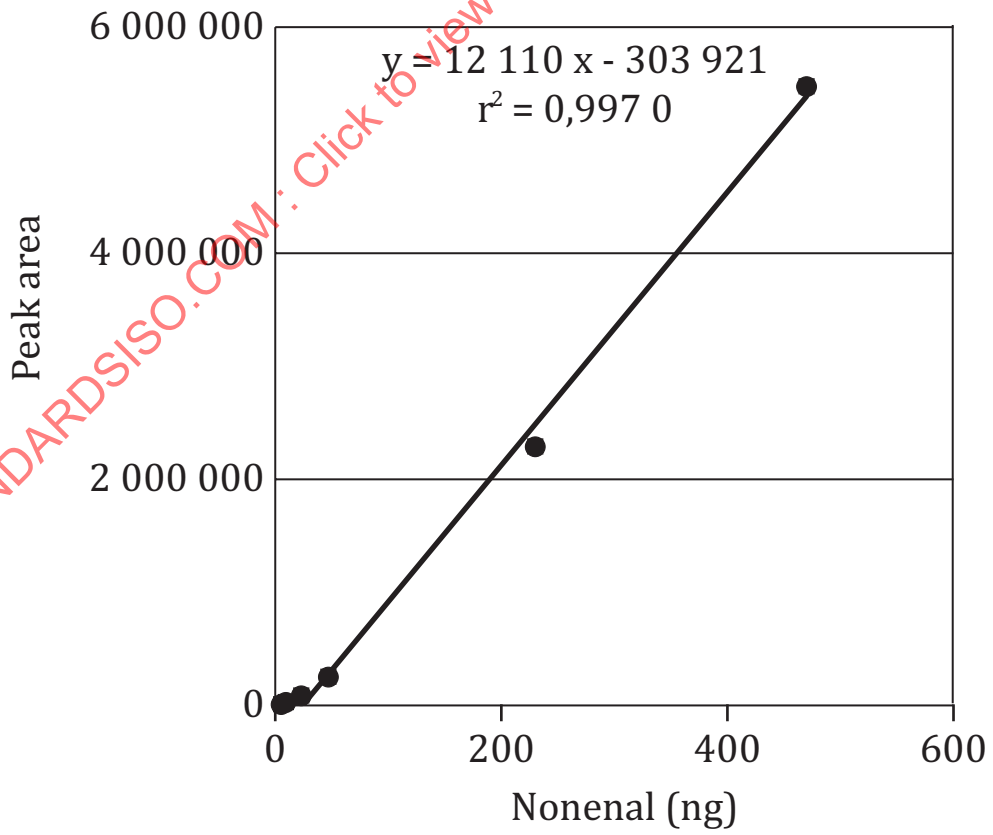


Figure A.8 — Calibration curve for nonenal (23 ng~470 ng)

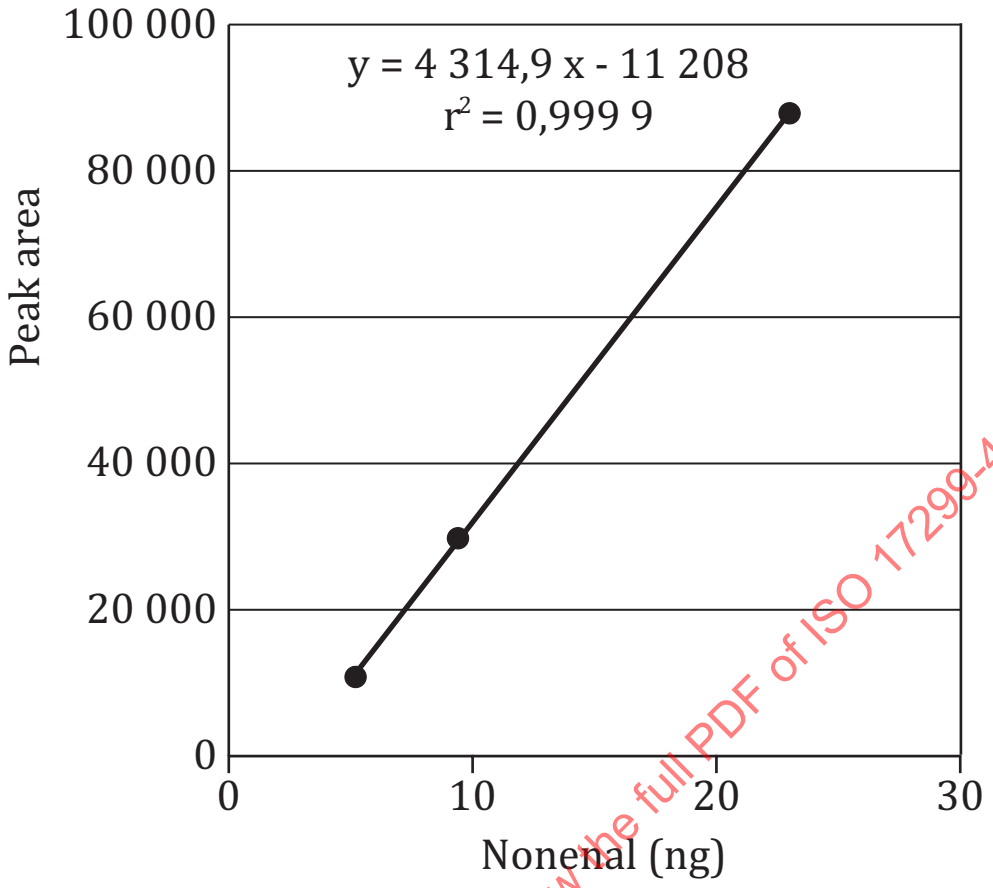


Figure A.9 — Calibration curve for nonenal (5,2 ng~23 ng)

Annex B
(informative)

Actual application of ion chromatography to the measurement of hydrogen sulfide

B.1 General

The analytical instruments have a wide variety in the kind of type or specification among the laboratories, so the measurement conditions are unique to each instrument, even for the measurement of the same odour chemicals. To make a convenience for users, the example of a typical set of measurement conditions and the results are shown below as a reference.

B.2 Analytical condition for Ion chromatography

Instrument	DX-500[a]
Injection volume	500 µl
Flow rate	1,5 ml/min
Eluent	2,5 mM Na_2CO_3/0,5mM $NaHCO_3$
Column	IonPac®[c] AS 12A[b](4 mm x 200 mm)
Column temperature	30 °C
Detector	Electric conductivity detector

[a] Dionex, general ion chromatograph with suppressor.

[b] Dionex, anion-exchange column for common inorganic anions and oxyhalides.

[c] IonPac® 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."

See Figure B.1 and Figure B.2.

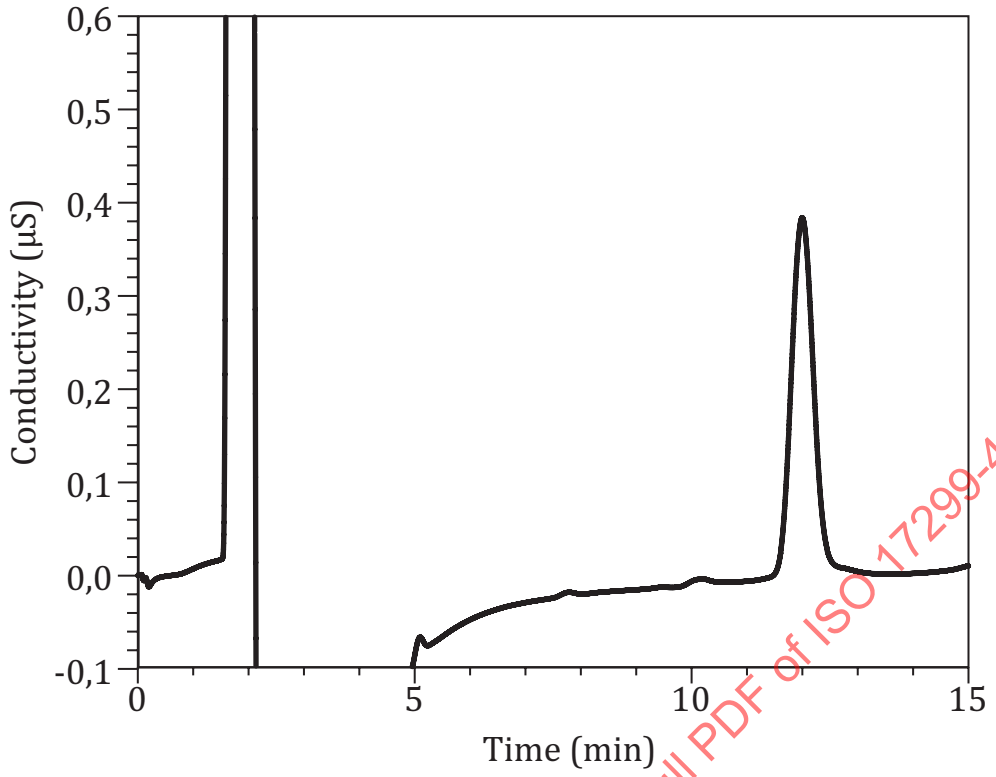


Figure B.1 — Chromatogram of sulfate ion standard solution (100 ng/ml)

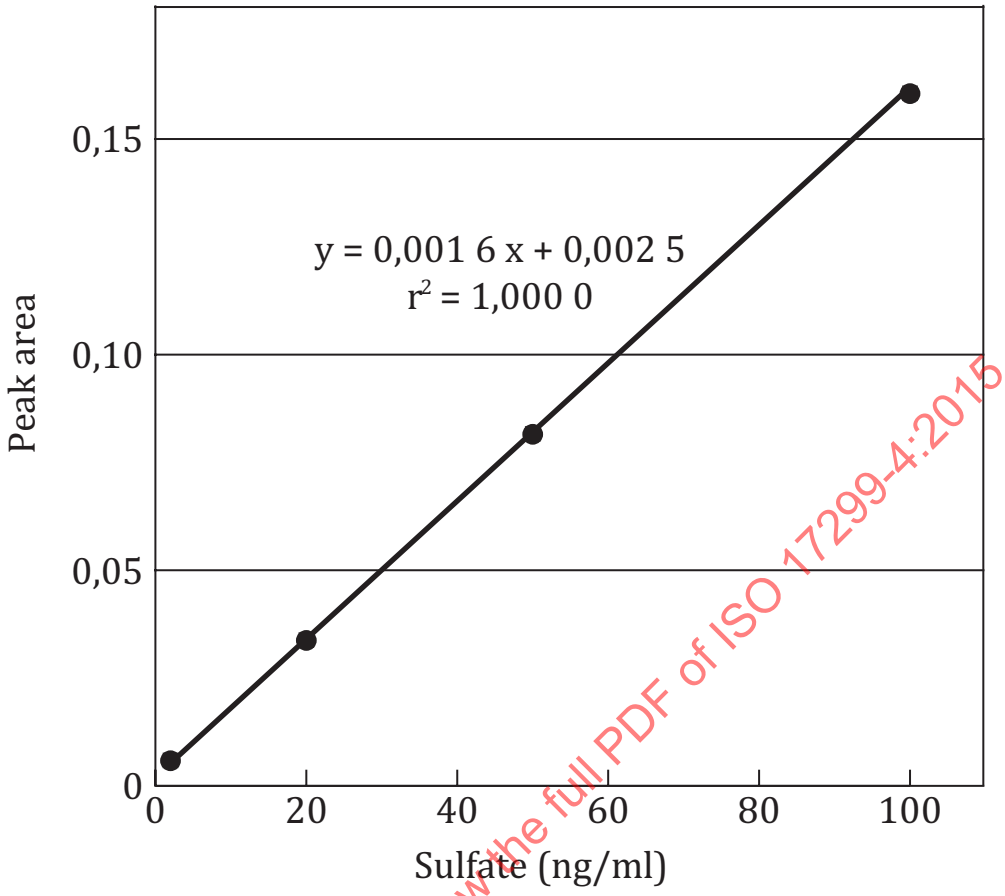


Figure B.2 — Calibration curve for sulfate ion

B.3 Hydrogen sulfide gas concentration

The formula which computes hydrogen sulfide gas concentration from sulfate ion concentration is shown below.

$$c(H_2S) = \frac{c_S \times \frac{M(H_2S)}{M(SO_4)} \times V_{abs}}{V_{gs}} \times \frac{V_{ideal}}{M(H_2S)} = \frac{c_s \times V_{abs}}{V_{gs}} \times k \quad \text{(B.1)}$$

where

$c(H_2S)$ is the hydrogen sulfide concentration of residual gas, in µl/l;

$M(H_2S)$ is the molecular weight of H_2S, i.e 34,09;

$M(SO_4)$ is the molecular weight of SO_4, i.e. 96,07;

V_{ideal} is the volume of ideal gas per 1 mol;

c_S is the sulfate ion concentration in the absorbing solution, in ng/ml;

V_{abs} is the volume of the absorbing solutions (ml) = 20;

V_{gs} is the volume of gas suction (l) = 2;

k is the conversion factor (ng/l →µl/l) = (34,09/96,07) × (24,287/34,09) × 10^{-3} = 2,528 × 10^{-4}.

Volume per ideal gas 1 mol: 24,287 (l, 23,5 °C).

0,02 M KOH/0,1 % H_2O_2 solution is recommended as the absorbing solution.

0,2 l/min of the gas suction flow rate is recommended.