भारतीय मानक

Indian Standard

IS 18624 : 2024
ISO 21330 : 2018

सिगरेट — सिगरेट के मेनस्ट्रीम स्मोक में वाष्पशील कार्बनिक यौगिकों का निर्धारण — जीसी/एमएस पद्धति

Cigarettes — Determination of Selected Volatile Organic Compounds in the Mainstream Smoke of Cigarettes — Method Using GC/MS

ICS 65.160

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Tobacco and Tobacco Products Sectional Committee, FAD 04

NATIONAL FOREWORD

This Indian Standard which is identical to ISO 21330 : 2018 ‘Cigarettes — Determination of selected volatile organic compounds in the mainstream smoke of cigarettes — Method using GC/MS’ issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standard on recommendation of the Tobacco and Tobacco Products Sectional Committee and approval of the Food and Agriculture Division Council.

The text of ISO standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words ‘International Standard’ appear referring to this standard, they should be read as ‘Indian Standard’; and

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to the following International Standards for which Indian Standards also exists. The corresponding Indian Standards which are to be substituted in their respective places, are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 3308 Routine analytical cigarette-smoking machine — Definitions and standard conditions	IS 16022 : 2015/ISO 3308 : 2012 Routine analytical cigarette-smoking machine — Definitions and standard conditions (*first revision*)	Identical
ISO 3402 Tobacco and tobacco products — Atmosphere for conditioning and testing	IS 16121 : 2023/ISO 3402 : 2023 Tobacco and tobacco products — Atmosphere for conditioning and testing (*first revision*)	Identical
ISO 8243 Cigarettes — Sampling	IS 12942 : 2018/ISO 8243 : 2013 Cigarettes — Sampling (*third revision*)	Identical

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Contents

Page

Introduction

The CORESTA (www.coresta.org) Special Analytes Sub-Group (since 2017 the Sub-Group changed its name to Smoke Analytes) carried out a collaborative study in 2005 to compare smoke analyte yield data obtained from different laboratories using their own preferred methodologies. This study have shown significant and unacceptable differences in volatiles yields, especially for 1,3-butadiene and acrylonitrile and suggested that further work was required to understand factors influencing the yield variability. Key parameters of existing methodologies have been reviewed and further studies have been carried out on selected volatiles between 2008[1] and 2009[2]. These studies investigated critical method steps that required optimization before incorporation into a CORESTA Recommended Method (CRM).

These studies have shown that similar yields were obtained when comparing data from Tedlar bag trapping with those from cooled impinger traps, the latter method being used by the majority of laboratories. It has been decided that the CRM would be based on collecting the selected volatiles from mainstream cigarette smoke in cryogenically cooled impinger traps containing methanol. The impinger solutions were fortified with benzene-D_6 and analysed by gas chromatography/mass spectrometry (GC-MS).

This document was produced after a 2009 collaborative study involving 20 laboratories from 12 countries using the ISO 3308 smoking regime[2]. Further data were provided for the same selected volatile substances from 10 samples with different tar yields from a 2012 collaborative study, which involved 16 laboratories from 11 countries[3]. The method includes recommendations about critical steps that should be controlled to provide data as robust and consistent as the repeatability and reproducibility data provided in the ISO standard. Statistical evaluations carried out according to ISO 5725-1 and ISO 5725-2 are included.

No machine smoking regime can represent all human smoking behaviour.

— It is recommended that cigarettes also be tested under conditions of a different intensity of machine smoking than those specified in this document.

— Machine smoking testing is useful to characterize cigarette emissions for design and regulatory purposes, but communication of machine measurements to smokers can result in misunderstandings about differences in exposure and risk across brands.

— Smoke emission data from machine measurements may be used as inputs for product hazard assessment, but they are not intended to be nor are they valid as measures of human exposure or risks. Communicating differences between products in machine measurements as differences in exposure or risk is a misuse of testing using ISO standards.

Indian Standard

CIGARETTES — DETERMINATION OF SELECTED VOLATILE ORGANIC COMPOUNDS IN THE MAINSTREAM SMOKE OF CIGARETTES — METHOD USING GC/MS

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of any other restrictions prior to use.

1 Scope

This document specifies a method for the quantification of selected volatile organic compounds (VOCs: 1,3-butadiene, isoprene, acrylonitrile, benzene and toluene) by GC-MS in mainstream cigarette smoke using ISO 3308 smoking parameters.

This method is applicable to cigarettes with nicotine-free dry particulate matter (NFDPM) yields between 1 mg/cigarette and 15 mg/cigarette.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3308, *Routine analytical cigarette-smoking machine — Definitions and standard conditions*

ISO 3402, *Tobacco and tobacco products — Atmosphere for conditioning and testing*

ISO 8243, *Cigarettes — Sampling*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

Selected volatiles are collected by passing the mainstream smoke of cigarettes through a glass fibre filter pad as specified in ISO 3308 (e.g. Cambridge filter pad, CFP) into cryogenic traps containing methanol.

The impinger solutions are fortified with internal standard and analysed by GC-MS.

5 Apparatus

The usual laboratory apparatus for use in preparation of samples, solutions and standards and, in particular, the following:

5.1 Smoking machine, complying with ISO 3308.

5.2 Impinger trapping system, capable of being connected in series, a cryogenically cooled liquid impinger to efficiently trap volatile organic compounds present in the vapour phase of mainstream smoke.

5.3 GC-MS system, to obtain chromatographic data to quantify specific ion (Selected Ion Monitoring mode, SIM or equivalent).

The GC shall be configured to perform split injections on a capillary column.

It is recommended to equip the GC with an auto sampler for sample injection.

5.4 Gas tight syringes, of appropriate volumes.

5.5 Fused silica capillary column, for example DB-624, length 60 m with internal diameter of 0,25 mm and 1,4 µm film thickness, or equivalent.

5.6 Spectrophotometer, to estimate 1,3-butadiene concentration in secondary stock solution.

6 Reagents

6.1 Dry ice.

6.2 Isopropanol, for Dewar flasks.

6.3 Methanol, HPLC grade or better.

The methanol should be checked to ensure the background levels of the analytes will not negatively affect the analysis.

6.4 Ethanol, reagent grade or equivalent.

6.5 Benzene-D_6, min. 99 % D; checked for the absence of not-labelled analogue.

6.6 Toluene-D_8, min. 99 % D; checked for the absence of not-labelled analogue.

6.7 1,3-Butadiene, min. 99 %.

6.8 Isoprene, min. 99 %.

6.9 Acrylonitrile, min. 99 %.

6.10 Benzene, min. 99 %.

6.11 Toluene, min. 99 %.

7 Preparation

7.1 Preparation of glassware

Glassware shall be cleaned and dried in such a manner which ensures that contamination from glassware does not occur.

It is important that all possible sources of contamination are removed from the work area.

7.2 Preparation of standards

7.2.1 General

Where available, certified reference solutions of the required standards and internal standards can be used.

7.2.2 Preparation of internal standard spiking solution

7.2.2.1 Internal standard stock solution

Transfer the contents of a 1 g ampoule of benzene-D_6 into a 10 ml amber volumetric flask. Dilute to volume with methanol.

The inclusion of other internal standards, such as toluene-D_8, may also be suitable. Laboratories shall demonstrate the suitability of the inclusion of additional internal standards.

7.2.2.2 Internal standard spiking solution

Using a volumetric pipette, transfer 4 ml of the stock solution (7.2.2.1) into a 100 ml volumetric flask and dilute to volume with methanol. This solution has a concentration of 4 000 µg/ml.

7.2.2.3 Storage

Store the diluted solutions in 25 ml vials with PTFE-lined caps in freezer.

7.2.3 Preparation of working standards for isoprene, acrylonitrile, benzene, and toluene

7.2.3.1 Primary isoprene, acrylonitrile, benzene and toluene stock solutions

Using gas tight syringes, weigh accurately 100 mg of isoprene, acrylonitrile, benzene, and toluene into separate 10 ml amber volumetric flasks that are half filled with methanol. Dilute each compound to volume with methanol. Each solution has a nominal concentration of 10 mg/ml.

NOTE Approximate volumes corresponding to 100 mg are: isoprene = 150 µl, acrylonitrile = 140 µl, benzene = 130 µl, toluene = 120 µl.

7.2.3.2 Secondary stock solution (mixture of isoprene, acrylonitrile, benzene and toluene primary stock solutions)

A combined secondary stock solution is prepared by transferring appropriate amounts (Table 1) of isoprene, acrylonitrile, benzene, and toluene primary stock solutions (7.2.3.1) into a 50 ml volumetric flask that is a third full with methanol. Dilute to volume with methanol.

Table 1 — Preparation of secondary stock solution

Analyte	Volume of primary stock (ml)	Concentration (µg/ml)
Isoprene	3,0	600
Acrylonitrile	1,0	200
Benzene	1,0	200
Toluene	1,0	200

7.2.3.3 Calibration standard solutions (for isoprene, acrylonitrile, benzene and toluene)

Prepare seven working standard solutions by mixing appropriate volumes of secondary stock solution (7.2.3.2) and internal standard spiking solution (7.2.2.2) to cover the concentration range of interest, i.e. (12 – 600) µg/ml (isoprene); (4 – 200) µg/ml (acrylonitrile); (4 – 200) µg/ml (benzene); (4 – 200) µg/ml (toluene) and 40 µg/ml of internal standard (e.g. benzene-D_6).

The concentration change of the highest calibration standard solution after adding internal standard solution should not be significant.

Transfer aliquots of each calibration standard solution into amber GC vials and fill each vial up to the shoulder of the vial to minimize headspace.

Adjust standard concentrations accordingly to reflect levels of volatiles found in smoke samples.

PTFE lined GC vial caps are recommended, although other materials may also be suitable.

7.2.3.4 Storage

Store all calibration standard solutions in freezer.

7.2.4 Preparation of working standards for 1,3-butadiene

7.2.4.1 Primary 1,3-butadiene stock solution

Attach a piece of chemically resistant polymer tubing to the valve of a cylinder containing 1,3-butadiene. Place a Pasteur pipette on the other end of the tubing and immerse the tip of the pipette into a 100 ml amber glass volumetric flask containing methanol up to the base of the neck of the flask. Open the valve and gently bubble the 1,3-butadiene into the methanol for approximately 5 min. Dilute to volume using methanol and mix well.

7.2.4.2 Secondary 1,3-butadiene stock solution

Pipette 1 ml of the primary 1,3-butadiene stock solution (7.2.4.1) into a 100 ml volumetric flask and dilute to volume with methanol. Mix well.

7.2.4.3 Determination of secondary 1,3-butadiene stock concentration

Pipette 1 ml of the secondary 1,3-butadiene stock solution (7.2.4.2) into a 100 ml volumetric flask and dilute to volume using ethanol. This solution is used only to check the concentration of the secondary stock solution and shall not be used to prepare the working standards.

Measure the absorbance of the solution against an ethanol blank on a spectrophotometer (use 1 cm long cuvettes). Conduct a wave scan from 200 nm to 250 nm to determine the wavelength of maximum absorbance. 1,3-butadiene in hexane absorbs at 217 nm whereas 1,3-butadiene in ethanol can have a peak shift. Measure the absorbance at the peak maximum.

Repeat the above measurement three more times and calculate the mean absorbance, A (at least three significant figures). The absorbance should be between 0,2 and 0,6 extinction units. If it is higher, make a new secondary stock solution using a smaller volume of the primary stock solution and repeat the spectrophotometer measurements to determine the concentration of the secondary stock. If the absorbance is lower, make a new secondary stock solution using a larger volume of the primary stock solution and repeat the spectrometer measurements to determine the concentration of the secondary stock.

The concentration of the secondary stock solution, in micrograms per millilitre, c_{2S}, is calculated by Formula (1):

$$c_{2S} = \frac{A}{20\,893} \times 54 \times 100 \times 1\,000 \quad (1)$$

where

A	is the mean absorbance;
20 893 l mol^{-1} cm^{-1}	is the molar absorption coefficient of 1,3-butadiene;
54 g/mol	is the molar mass of 1,3-butadiene;
100 ml	is the volume of the solution;
1 000	is the units conversion factor.

7.2.4.4 1,3-butadiene calibration standard solutions

Prepare five working standard solutions by mixing 1,3-butadiene secondary stock solution (7.2.4.2) and internal standard spiking solution (7.2.2.2) that cover the concentration range of interest, i.e. (5 to 50) µg/ml for 1,3-butadiene and 40 µg/ml for internal standard.

Transfer aliquots of each calibration standard solution into amber GC vials and fill each vial up to the shoulder of the vial to minimize headspace.

NOTE Certified concentrations of 1,3-butadiene in methanol can be purchased and used to prepare the standards.

7.2.4.5 Storage

Store all calibration standard solutions in freezer until use.

Stability of all standard solutions for all components should be assessed by laboratories under their own in-house conditions before use as stability is dependent on the actual storage conditions of each laboratory.

8 Sampling

Carry out sampling in accordance with ISO 8243.

9 Tobacco product preparation

Condition the cigarettes in accordance with ISO 3402.

10 Sample generation — Smoking of cigarettes

10.1 General

The smoking parameters for which the method has been studied are defined in ISO 3308 (see Table 2).

Table 2 — Smoking parameters

Smoking regime	Puff volume (ml)	Puff frequency (s)	Puff duration (s)	Ventilation blocking (%)
ISO 3308	35	60	2	0

10.2 Smoking machine setup

An analytical cigarette-smoking machine complying with the requirements of ISO 3308 is required.

A methanol-filled impinger system is required that efficiently traps the VOCs of interest. An example using two impingers is provided in Figure 1; however, other trapping systems using a different number of impingers, different impinger tip styles (capillary, fritted, etc.) and a different volume of trapping solution can also provide suitable trapping efficiency.

Fill all coolant reservoirs with one-third full of isopropanol. Add dry ice until each reservoir is filled halfway. The number of reservoirs required is dependent on the impinger design and has to be optimized to ensure that all volatiles are trapped efficiently.

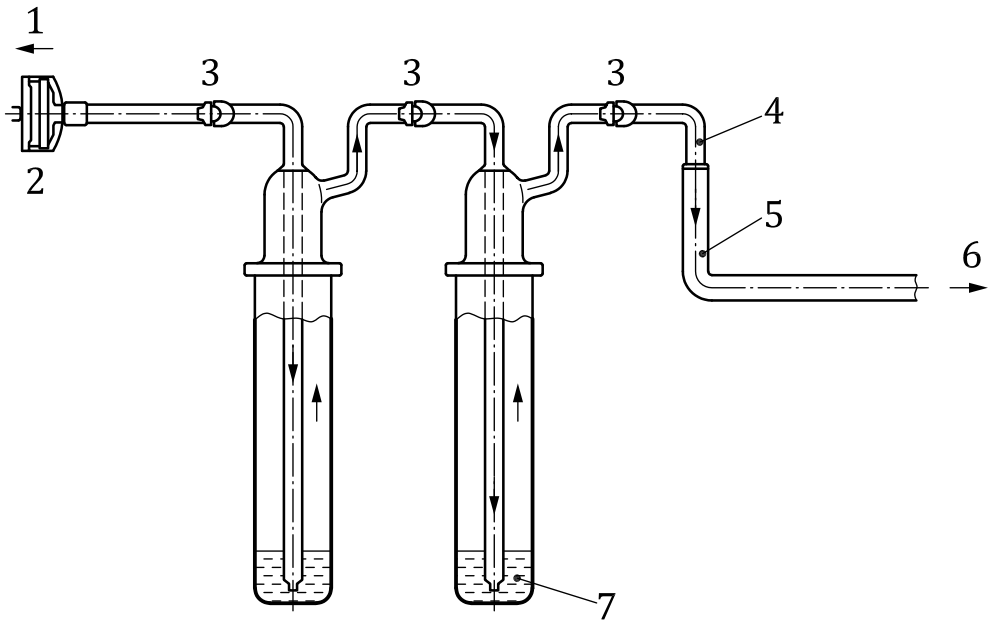
Add 10 ml of methanol to each impinger and place the impingers into the coolant reservoir containing the dry ice/isopropanol solution. Check each coolant reservoir to ensure that the temperature is at or below –70 °C.

A volume other than 10 ml of methanol may need to be added to each impinger depending on the particular style of impinger used.

The impingers shall be given sufficient time to cool to –70 °C or below before starting smoke collection.

Connect the impingers to the smoking machine (see Figure 1).

Check and adjust the puff volume drawn by the smoking machine at all channels.



Key

1 to smoking machine
2 glass fibre filter pad
3 glass ball joint or tubing connections
4 glass elbow or tubing
5 tubing
6 to syringe
7 100 ml impingers each containing 10 ml methanol

Figure 1 — Example of an impinger setup for smoking machines

To determine whether a leak has occurred in the smoking machine impinger setup, use a leak tester. If the fluid column does not maintain its position but drops, then there is a leak in the system.

It is recommended that tubing other than silicone tubing is used for connections between the smoking machine and the impingers (i.e. polyethylene, polyvinyl chloride, polypropylene). Methyl silicone tubing is not recommended since adsorption of the analytes can occur. Tubing should be as short as possible to minimize the potential for any adsorption.

It is recommended that the trapping efficiency is checked when validating this method. To check the trapping efficiency of the method, add an additional impinger and follow the method accordingly. Analyse each impinger individually for the volatile compounds of interest. If no VOCs are detected in the additional impinger then only the prescribed number of impingers is required to trap all the VOCs effectively. Poor trapping efficiency may be due to the impinger or impinger tip design.

If a carryover occurs, it is a responsibility of each laboratory to assess the carryover with respect to the specific trapping system design and decide how to manage it. Carryover should be repeatable, less than 5 % (ideally less than 1 %) and if greater than 5 %, should be reported or corrected in calculations.

10.3 Smoking

10.3.1 General

The cigarettes are smoked according to ISO 3308 (see Table 2) with the following modifications.

10.3.2 Linear smoking

Five or 10 cigarettes are smoked per trap.

If 10 cigarettes are smoked, then the CFP needs to be changed after five cigarettes to avoid total particulate matter (TPM) breakthrough.

10.3.3 Rotary smoking

Ten cigarettes are smoked per trap onto 92 mm CFP.

11 Sample analysis

11.1 Preparation of sample

After all samples have been smoked, the TPM shall be determined on the CFP as a quality control measure and the CFP discarded. Laboratories should evaluate the trapping system for losses in the tubing that connects the pad holder to the impinger(s) and the connections between impingers (if more than one impinger is used). If there are losses, the tubing may be rinsed or extra clearing puffs may be taken. The impingers shall be kept in the cooling reservoir until sampling is complete.

After all samples have been smoked following ISO 3308, each impinger is spiked with 100 µl of internal standard spiking solution. The impingers are stoppered and vortexed to ensure that the extract is well mixed. If the impinger setup requires more than one impinger then the trapping solutions are combined in such a way as to ensure complete mixing of both impingers. Transfer an aliquot of the impinger solution into an amber GC vial for GC-MS analysis. Fill each vial up to the shoulder of the vial to minimize headspace and cap tightly. Prepare all samples in duplicate and keep a set in the freezer in case repeated analysis is required.

Samples are stable when stored in the freezer (temperature below –20 °C) for a maximum of 48 h.

It is recommended that sample stability is determined under storage conditions when validating this method.

11.2 Determination

11.2.1 GC-MS operating conditions

Set up and operate the GC-MS system in accordance with the manufacturer's instruction.

The following parameters have been found to be suitable for separation.

GC parameters:

Injector temperature:	150 °C
Column temperature:	40 °C (6 min) 20 °C/min to 225 °C (6 min)
Carrier gas:	Helium
Carrier gas flow:	1,5 ml/min (1,6 bar)
Injection mode:	Split
Injection split ratio:	30:1
Injection split flow:	30 ml/min
Injection volume:	3 µl

MS parameters:

Transfer line temperature:	240 °C
Source temperature:	240 °C
Acquisition mode:	SIM (or SCAN)
Solvent delay:	Column dependent
Low mass:	40,0
High mass:	200,0

Ion traces (m/z):	**Quantification**	**Confirmation**
1,3-butadiene	54	53
Isoprene	67	68
Acrylonitrile	52	53
Benzene	78	77
Benzene-D_6	84	83
Toluene	91	92

Chromatographic separation should be similar to example chromatograms shown in Figures A.1 to A.4.

NOTE The choice of chromatographic conditions and data acquisition parameters can differ for different instrument configurations.

11.2.2 Calibration

Analyse, successively, each working standard solution (7.2.3.3 and 7.2.4.4) by GC-MS. Record the area of each of the analysed compounds and the internal standard peaks. Generate a calibration curve for each of the compounds by calculating a linear regression equation of the peak area ratios of the analysed compounds to the internal standard against their concentration. The intercept of these regression lines should be close to zero.

11.2.3 Calculation

The yield of individual selected volatile compounds in the mainstream smoke of cigarettes, m_i, expressed in micrograms per cigarette, is given by Formula (2):

$$m_i = \frac{C_i V}{N_{cig}} \quad (2)$$

where

C_i is the concentration of the analyte, in micrograms per millilitre, in the sample;

V is the volume, in millilitres;

N_{cig} is the number of cigarettes smoked.

The expression of the laboratory data depends on the purpose for which the data are required, and the level of laboratory precision. Any further statistical analyses should be calculated and expressed on the basis of the laboratory data before any rounding has taken place.

The yield of individual selected volatile compounds in the mainstream smoke of cigarettes is reported in micrograms per cigarette (µg/cig) to the nearest 0,1 µg.

12 Repeatability and reproducibility

12.1 General

An international collaborative study was conducted in 2012 involving 16 laboratories and 10 cigarette samples. This provided data on the measurement of the same selected volatiles substances in five replicate analyses of 10 samples (see Table 3) performed under ISO smoking regime. The values for repeatability, *r*, and reproducibility, *R*, given in Tables 4 to 8, were obtained using this method. The statistical evaluation was performed according to ISO 5725-2.

Table 3 — Cigarette test samples of 2012 the collaborative study

Sample	Product characterization	NFDPM yield (mg/cigarette)
Sample 1	Dark air-cured product	10
Sample 2	American blended product	8
Sample 3	American blended product	6
Sample 4	Virginia blended product	4
Sample 5	Virginia blended product	2
Sample 6	Virginia blended product	10
Sample 7	Charcoal filtered/blended product	1
3R4F	Kentucky Reference 3R4F/American blend	8
1R5F	Kentucky Reference 1R5F/American blend	2
CM6	CM6 Test piece/Virginia blend	15

12.2 Results of the 2012 collaborative study

Calculated statistical data for the individual selected volatile organic compounds are given in Tables 4 to 8.

Table 4 — 1,3-Butadiene

Sample	NFDPM yield (mg/cigarette)	N[a]	Mean	*r*	*R*
			(µg/cigarette)		
CM6	15	16	61,1	12,2	34,6
1R5F	2	16	12,1	3,3	7,4
3R4F	8	17	40,5	9,1	19,9
1	10	13	31,0	6,9	16,2
2	8	13	39,0	10,1	17,8
3	6	12	35,1	7,5	16,2
4	4	12	18,0	4,1	7,8
5	2	13	10,1	2,7	4,3
6	10	13	46,1	11,7	21,6
7	1	12	8,2	2,4	4,5

[a] *N* = number of data sets taken for statistical analysis after removal of outliers.

Table 5 — Isoprene

Sample	NFPDM yield (mg/cigarette)	N[a]	Mean	*r*	*R*
			(µg/cigarette)		
CM6	15	16	563,9	89,5	246,1
1R5F	2	16	117,8	26,0	66,9
3R4F	8	16	349,3	65,3	124,6
1	10	13	176,2	45,0	116,6
2	8	13	278,9	70,6	143,3
3	6	11	271,6	36,8	137,2
4	4	13	150,1	39,3	85,1
5	2	12	65,3	13,3	28,8
6	10	13	278,4	50,9	182,4
7	1	13	63,1	15,4	40,6

[a] *N* = number of data sets taken for statistical analysis after removal of outliers.

Table 6 — Acrylonitrile

Sample	NFDPM yield (mg/cigarette)	N[a]	Mean	*r*	*R*
			(µg/cigarette)		
CM6	15	15	12,3	2,1	6,1
1R5F	2	14	2,1	0,7	1,7
3R4F	8	17	8,5	2,5	4,1
1	10	13	10,2	2,3	5,1
2	8	13	7,9	1,8	3,6
3	6	12	5,8	1,5	2,8
4	4	11	2,2	0,5	1,6

[a] *N* = number of data sets taken for statistical analysis after removal of outliers.

Sample	NFDPM yield (mg/cigarette)	N[a]	Mean	*r*	*R*
			(µg/cigarette)		
5	2	12	1,3	0,3	1,3
6	10	13	7,9	1,8	4,3
7	1	11	1,0	0,3	1,3

[a] *N* = number of data sets taken for statistical analysis after removal of outliers.

Table 7 — Benzene

Sample	NFDPM yield (mg/cigarette)	N[a]	Mean	*r*	*R*
			(µg/cigarette)		
CM6	15	15	60,4	7,6	25,7
1R5F	2	16	13,9	2,9	7,5
3R4F	8	16	41,6	8,1	18,6
1	10	13	34,5	5,9	15,4
2	8	12	37,3	6,1	16,4
3	6	11	32,9	4,2	14,8
4	4	13	17,1	4,2	9,4
5	2	13	10,5	2,5	6,6
6	10	13	41,0	7,2	18,0
7	1	13	7,5	2,0	7,0

[a] *N* = number of data sets taken for statistical analysis after removal of outliers.

Table 8 — Toluene

Sample	NFDPM yield (mg/cigarette)	N[a]	Mean	*r*	*R*
			(µg/cigarette)		
CM6	15	15	87,3	12,9	34,9
1R5F	2	15	19,2	5,1	11,7
3R4F	8	16	67,4	16,4	33,5
1	10	12	59,7	11,6	28,1
2	8	12	58,8	10,6	27,8
3	6	11	47,1	9,9	21,7
4	4	12	23,9	5,3	13,3
5	2	12	14,3	3,9	8,9
6	10	12	55,9	10,6	25,6
7	1	12	9,7	4,0	9,7

[a] *N* = number of data sets taken for statistical analysis after removal of outliers.

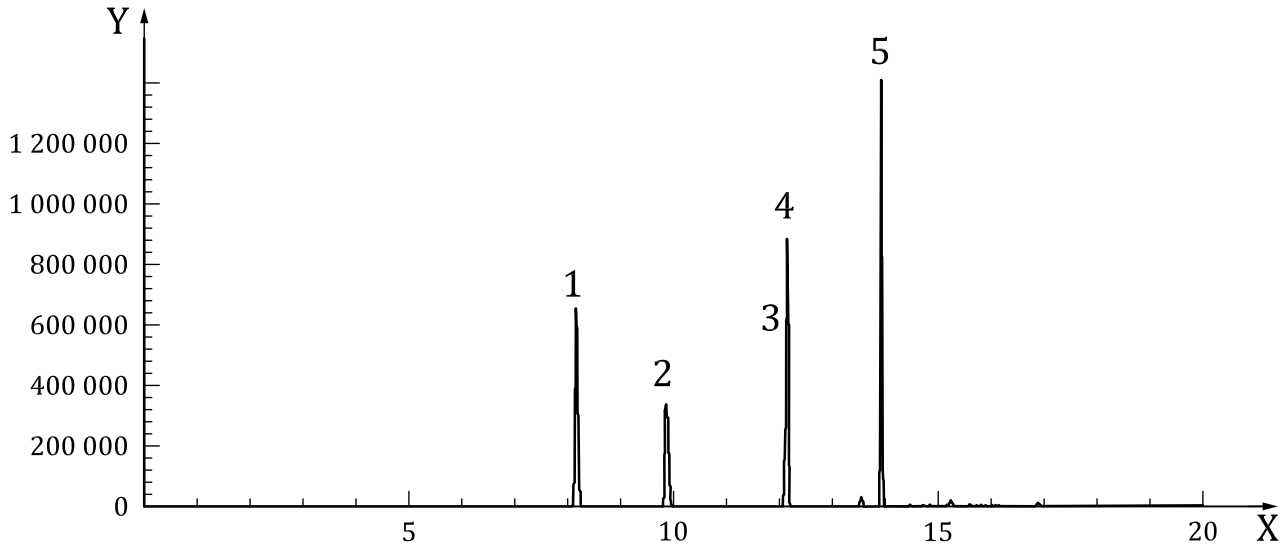
13 Test report

The test report shall state all tested product(s) each with unique identification, reference to the smoking regime used for sample generation, the yield of selected VOCs in micrograms per cigarette smoked, and the method used. The test report shall include all conditions and deviations which can affect the result. All information should be recorded in fully traceable manner.

Annex A
(informative)

Examples of chromatograms

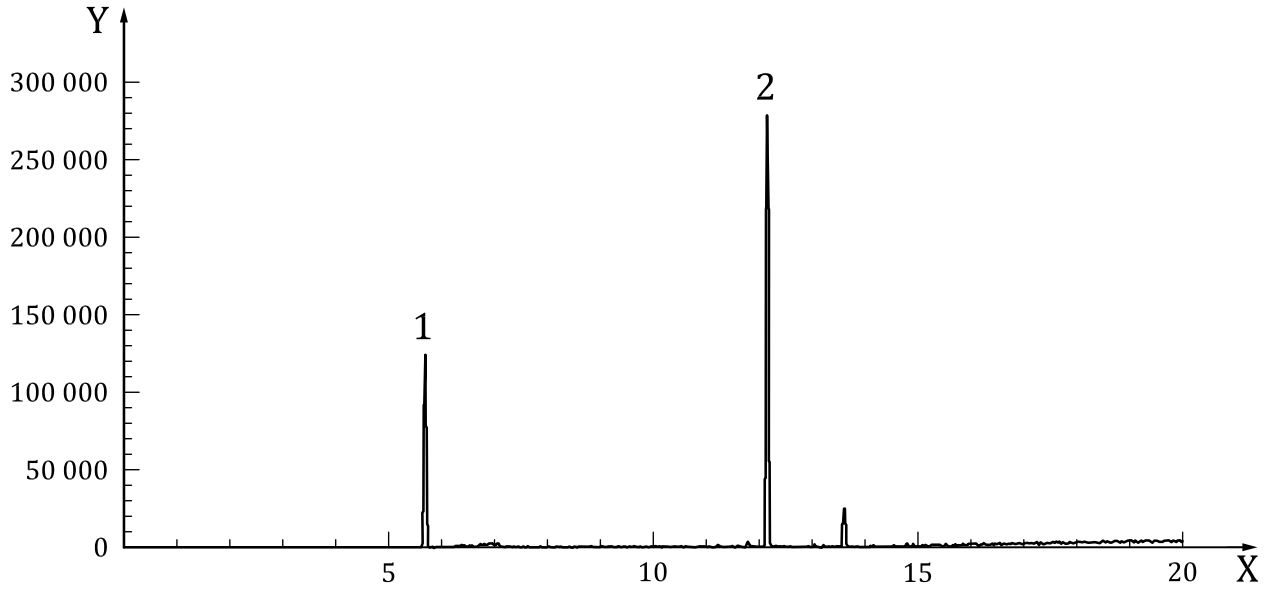
Example chromatograms are given in Figures A.1 to A.4.



Key

X time, min
Y abundance
1 isoprene
2 acrylonitrile
3 D_6-benzene
4 benzene
5 toluene

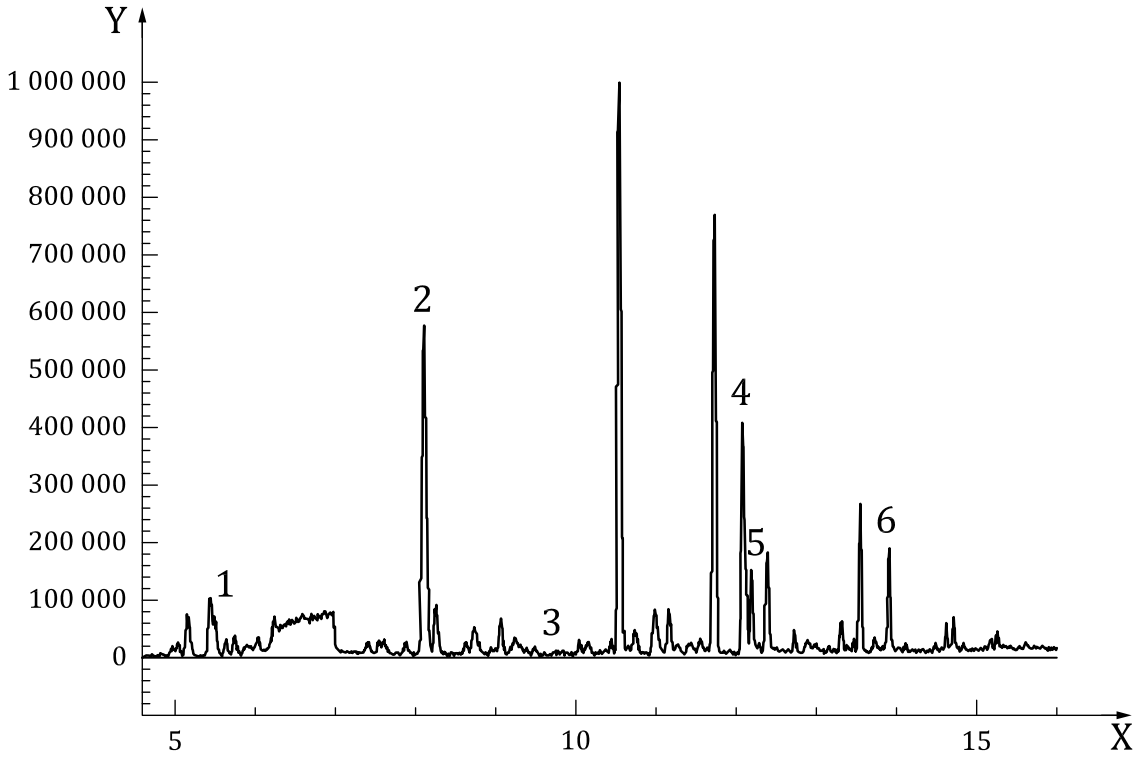
Figure A.1 — Example of a chromatogram of toluene, isoprene, benzene and acrylonitrile calibration standard (full scan mode)



Key

X	time, min	1	1,3-butadiene
Y	abundance	2	D_6-benzene

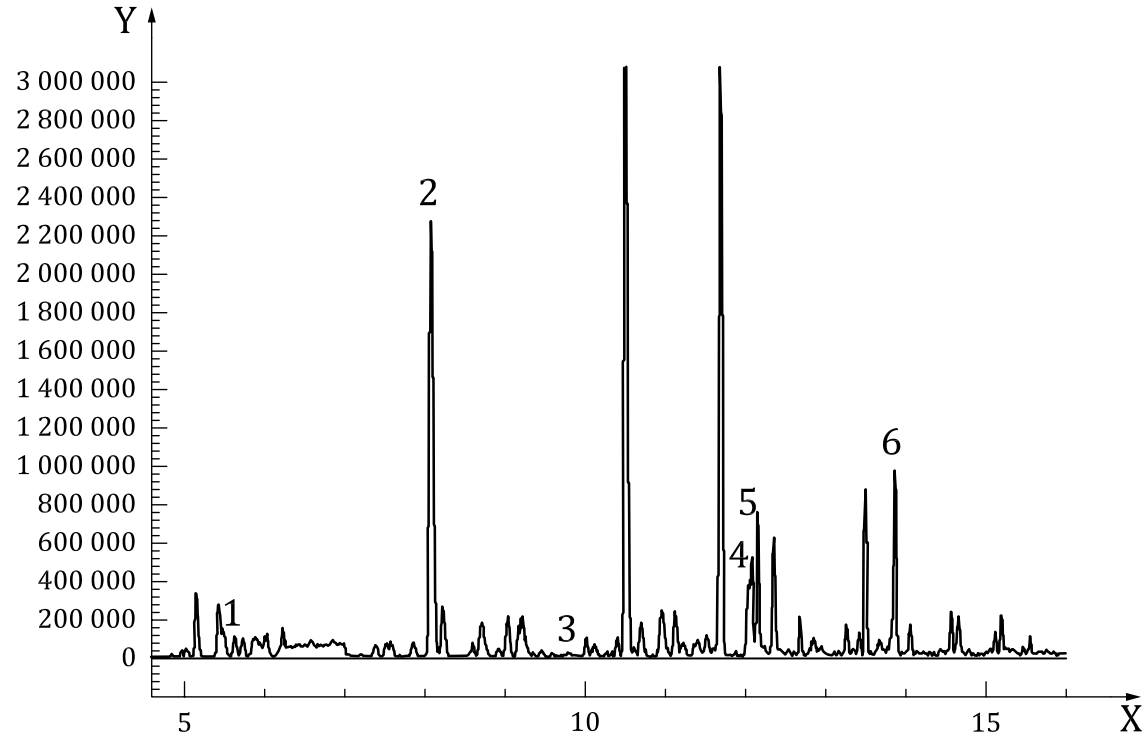
Figure A.2 — Example of a chromatogram of a 1,3-butadiene calibration standard (full scan mode)



Key

X	time, min	3	acrylonitrile
Y	abundance	4	D_6-benzene
1	1,3-butadiene	5	benzene
2	isoprene	6	toluene

Figure A.3 — Example of a 1R5F chromatogram (full scan mode)



Key

X	time, min	3	acrylonitrile
Y	abundance	4	benzene
1	1,3-butadiene	5	D_6-benzene
2	isoprene	6	toluene

Figure A.4 — Example of a 3R4F chromatogram (full scan mode)

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