Danish Society of Indoor Climate

Standard Test Method

for

Determination of the Indoor-Relevant Time-Value

by Chemical Analysis and Sensory Evaluation

2nd Edition
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1 Foreword

The 1st edition of the present criteria document was published in 1994 by the Danish Society of Indoor Climate (formerly the Danish Indoor Climate Labelling Association). The concept of the Labelling Scheme was developed by the Danish Building and Urban Research Institute and the National Institute of Occupational Health in Denmark.

In addition to the present criteria document describing the standard test method and calculation of the indoor-relevant time-value by chemical analysis and sensory evaluation, other criteria documents are issued by the Danish Society of Indoor Climate:

- Standard Test Method for Determination of Emission of Particles
- General Labelling Criteria
- Specific testing and labelling criteria for individual product areas

The board of the Danish Society of Indoor Climate has approved this criteria document after a hearing in Denmark and Norway.

2 Introduction

The Danish Indoor Climate Labelling Scheme is based on evaluations of the impact on human health and comfort of individual volatile organic compounds emitted by materials. The emissions are evaluated in terms of their possible cause of odour and irritation of mucous membranes in the upper airways. At the same time, the product must not emit substances that are known[1] to cause carcinogenic effects in humans.

The results of the emission tests are presented in terms of an indoor-relevant time-value. The time-value summarises all emission test results in one single figure, which gives the time (in days) until the concentration of all individual substances emitted from a product in a standard room is below half the threshold value for odour and mucous irritation. The indoor-relevant time-value is based on results of the chemical analyses as well as the sensory evaluations.

The Labelling Scheme has objectives such as:

- to promote the development of the indoor air friendly products by providing manufacturers with emission data
- to provide architects, projecting engineers, and end users with easy-to-understand emission data

The present criteria document describes how the emission test results should be evaluated according to the Danish Society of Indoor Climate.

Today, the test chamber methods and procedures are set by the following European standard documents:

- CEN ENV 13419 Building products – determination of the emission of volatile organic compounds – Part 1: Emission test chamber method

[1] According to the International Agency for Research on Cancer (IARC, WHO): "Overall Evaluations of Carcinogenicity to Humans, Group 1: Carcinogenic to humans"

- CEN ENV 13419 Building products – determination of the emission of volatile organic compounds – Part 2: Emission test cell method
- CEN ENV 13419 Building products – determination of the emission of volatile organic compounds – Part 3: Procedure for sampling. Storage and preparation of test specimens

These standards are considered the primary reference documents of emission testing for the Danish Society of Indoor Climate, unless other requirements are prescribed by the present criteria document.

A proper understanding and use of test chamber techniques is crucial for obtaining reliable results. The best proof of quality of the test laboratory is that the laboratory is accredited for the test methods or at least has validated the quality through inter-laboratory comparisons.

3 Scope

This standard specifies the test method for determination of the indoor-relevant time-value by emission tests of newly manufactured products according to the Danish Society of Indoor Climate.

The method includes both chemical analyses and sensory evaluations.

To evaluate the influence of a building product or interior product on the indoor air quality, this standard test method is used in addition to the specific product standard or test and labelling criteria.

Requirements in the product specific test and labelling criteria take priority over requirements given in this standard test method. The method can only be used as basis for labelling of a product, if the specific Product Standard or Test and Labelling Criteria is available.

4 Definitions, Symbols and Units

Acceptable indoor air quality, C_L [µg/m^3 or mg/m^3]: The acceptable concentration of a compound in the indoor air is half the lowest threshold value of odour and/or mucous membrane irritation.

Air exchange rate, n [1/h]: Ratio of the volume of clean air that ventilates the emission test chamber and the emission test chamber volume. This definition implies full mixing of the air in the test chamber.

Air velocity, v [m/s]: Speed of air passing over the surface of the test specimen.

Airflow rate, Q [m^3/h]: Volume of air entering the emission test chamber per time unit.

Determining compound: One or more chemical compound, which is determining for the time-value. That is the compound, which takes the longest time to decrease to a level below half of the threshold value for odour and/or irritation.

Emission constant, K [1/h]: Constant used to model the emission profile of a compound.

Emission test chamber concentration, C [µg/m^3]: Concentration of a specific compound measured in the emission test chamber.

Emission test chamber: Enclosure with controlled operational parameters. Emission test chambers are used for analyses and evaluations of the emissions from a product.

Headspace analysis: A method to determine which compounds may be emitted from a product.

Indoor-relevant time-value, t_L [days]: The time it takes to reach the acceptable indoor air quality for all emitted compounds in a standard room under standard conditions.

Irritation threshold value, IT [µg/m^3]: The lowest concentration at which the compound causes mucous membrane irritation.

Material loading factor, L [m^2/m^3]: Ratio of exposed surface area of the test specimen and the empty emission test chamber volume.

Odour threshold value, OT [µg/m^3]: The concentration of an odourant, which have a probability of 0.5 to be registered by a test panel (detection odour threshold).

Sensory evaluation: Panellists express their impression of the air quality by evaluating the acceptability and odour intensity of the air.

Specific emission rate, R [µg/(m^{2}h)]: Mass of a volatile organic compound emitted from a product per unit time and area.

Standard room: A fictive room used as a model for the calculation of indoor air concentrations.

Test specimen: A sample of the material specially prepared in order to simulate the emission behaviour of the material or product.

VOCBase: Reference database containing threshold values for odour and irritation of mucous membranes.

Volatile organic compounds, VOC: Organic compounds emitted from the test specimen in the range from n-hexane to n-hexadecane when sampled on Tenax and analysed with a gas chromatograph using a non-polar or slightly polar capillary column.

5 Test Principle

The principle of the test is to determine the indoor-relevant time-value based on the emission rate of VOCs emitted from a product.

The indoor-relevant time-value states the time (in days) until the concentration of all individual substances emitted from a product in a standard room is below half the threshold value for odour and mucous irritation. The time-value is based on results of the chemical analyses as well as the sensory evaluations. At the same time, the product must not emit substances that are known to cause carcinogenic effects in humans.

The emission test is performed in an emission test chamber at controlled indoor environmental conditions.

When the emission profile of a product is obtained, the indoor-relevant time-value can be calculated. The calculated time-value is controlled by sensory evaluation of the emissions.

6 Sampling, Transportation and Storage

The sampling of the material to be tested must be in accordance with the specific product standards. The production date must be given. Samples shall be taken at the manufacturer's, agent's or the importer's stock.

The sample shall be transported to the test laboratory in unbroken and original packaging. The date of arrival of the sample at the test laboratory shall be registered.

If the test procedure can not be started immediately at receipt of the sample, the sample must be stored in the original packaging, if airtight. If the original packaging is not air tight, the sample must be repacked in airtight, inert wrapping. The sample may be stored for a maximum of three weeks before starting the test procedure.

The material to be tested should at all times be stored and transported under conditions that do not influence the indoor environmental properties of the material, i.e. extreme temperature and humidity conditions.

7 Test Facilities and Conditions

7.1 Facilities for Chemical Emission Tests

Facilities designed and operated to determine product emissions of VOCs should at least include the following: emission test chamber, clean air supply and humidification system, air mixing system, monitor and control systems according to the specifications given in CEN ENV 13419-1, 5.1 through 7.6.

7.2 Facilities for Sensory Evaluations

A special test chamber, the CLIMPAQ (Nordtest, 1998), has been developed and designed for sensory evaluations of material emissions. The CLIMPAQ is made of glass and has a volume of 50.9 l. Other test chamber designs may be used for the sensory evaluations. The device to present the air to be evaluated by the sensory panel must, however, be designed according to the description given below. The device should be shaped as a funnel with the dimensions given in Table 1.

Table 1 Dimensions of funnel

Length	450 mm
Inside diameter at outlet	80 mm
Inside diameter at inlet	25 mm

The test chamber, including the funnel, should be made of a non-emitting, chemically inert and cleaning-friendly material. Thus, the test chamber may be made of e.g. polished stainless steel or glass.

For the sensory evaluations, it is important that the air extracted from the test chamber is not mixed with the ambient air. The above-mentioned funnel and a specified airflow rate through the funnel ensure this.

The required air flow rate through the funnel is min. 0.9 l/s. This should be taken into account when planning the test specimen area used for the test.

Outline of a test chamber for sensory evaluations of emissions is shown in Figure 1.

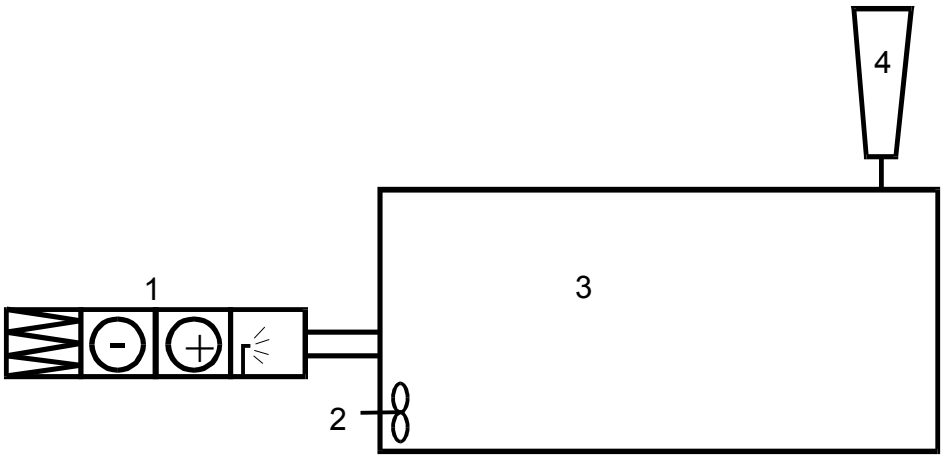

Figure 1 Outline of a test chamber for sensory evaluations of material emissions. 1: System to condition the supply air; 2: Mixing fan; 3: Test chamber space for placing test specimens; 4: Funnel providing air for sensory evaluation.

7.3 Test Conditions

The emission of VOCs from materials depends, among other factors, on the air temperature, relative humidity and air velocity. To obtain comparable and reproducible results, the emission test chambers used should be operated at specified conditions.

The test conditions should be controlled and maintained throughout the test period. All parameters should be within the ranges given.

The operating conditions of the test chambers should for both the chemical tests and sensory evaluations be kept at conditions identical to those of the standard room:

Air temperature	23°C ± 1°C
Relative humidity	50% RH ± 5% RH
Air velocity	0.2 m/s ± 0.1 m/s

8 Preparation of Test Specimens

Samples of the product to be tested may be cut in appropriately sized pieces to satisfy the analytical requirements.

Newly cut edges and backsides normally not exposed to the indoor air should be sealed tightly, e.g. with a low-emitting aluminium tape. If appropriate, and to avoid excess use of sealing materials, the samples may be arranged back-to-back with the edges tightly sealed.

All product samples must be prepared in accordance with the specific product standard or test and labelling criteria.

8.1 Material Load

8.1.1 Standard Room

The standard room has the characteristics given in Table 2:

Table 2 Characteristics of the standard room

Volume	17.42 m^3
Surface areas	
Floor/ceiling	7 m^2
Walls	24 m^2
Doors	2 m^2
Window frames	0.2 m^2
Sealants	0.2 m^2
Fixtures	4 m^2

The indoor environmental conditions in the standard room are given in Table 3.

Table 3 Indoor environmental conditions in the standard room

Air change rate	0.5 1/h
Air temperature	23°C
Relative humidity	50% RH
Air velocity	0.15 m/s

8.1.2 Chemical Emission Testing

When carrying out the chemical measurements, the area specific emission rate and the resulting concentration in the standard room may be calculated on basis of the actual test chamber volume, ventilation rate and material loading factor. Based on the material and type of emissions, the material load should be determined in order to be sufficient for the chemical analysis.

8.1.3 Sensory Evaluations

When performing the sensory evaluation, the air quality in the test chamber should be similar to that of the standard room. This is considered fulfilled when the area specific airflow rate is identical in the two spaces.

The area specific airflow rate is defined as:

$$q = Q/A = n/L \qquad \text{(Eq. 1)}$$

where

q: area specific airflow rate [l/(s m^2)]
Q: airflow rate [l/s]
A: area of test specimen surface [m^2]
n: air change rate [1/h]
L: loading factor [m^2/m^3]

The Equation given above results in loading factors and area specific airflow rates given in Table 4.

Table 4 Examples of area specific airflow rates in the standard room

Standard room	Surface area in standard room [m^2]	Area specific airflow rate in standard room, q [l/s m^2]	Desired surface area, A [m^2] in test chamber at Q = 0.9 l/s
Floor/ceiling	7	0.35	2.57
Wall	24	0.1	9.0
Door	2	1.21	0.74
Window frame	0.2	12.1	0.07
Sealant	0.2	12.1	0.07
Fixtures	4	0.6	1.5

The area specific airflow rate is calculated as the airflow rate divided by the surface area in question. The airflow rate in the standard room is: 17.42 m^3 x 0.5 h^{-1} = 8.71 m^3/h = 2.42 l/s.

9 Chemical Emission Testing

Emissions decrease as a function of time. The decay rate depends among other factors on the physical and chemical structure of the material, the temperature, relative humidity and air movement in the surrounding microenvironment.

9.1 Initial Analyses

The purpose of the initial analyses is to obtain information about, which compounds may possibly emit from the material. This is to ensure that the proper methods of analysis are used.

If the type of emission from the product is not known, the test programme is based on an initial qualitative analysis. The qualitative analysis may be a static headspace analysis, in which the test specimen is placed in an airtight container. The container is heated and after a suitable time, air samples are analysed by using gas chromatography combined with mass-spectrometric detection (GC-MS).

Alternatively, an initial analysis may be carried out by sampling the exhaust air from the test chamber 24 hours' after placing the test specimen in the chamber.

For most organic compounds, sampling on Tenax followed by a GC-MS analysis is suitable. Compounds like formaldehyde, ammonia and isocyanates should, however, be analysed by other methods.

9.2 Emission Test and Air Sampling

Before the test specimen is placed in the test chamber, air samples are taken from the empty chamber. These samples are used to document the background concentration of VOCs in the supply air and test chamber system.

The test specimen is placed in the test chamber and positioned in a way so that the emitting surface is parallel to the direction of the airflow in the chamber.

The exhaust airflow, or a special sampling outlet, may be used as the sampling port for chemical analyses.

9.3 Emission Test Duration

The duration of the emission test is determined by the specific emission from the product.

When testing materials with an unknown emission profile, air samples should at least be taken over a period of 28 days with a minimum of 3 samplings, e.g. after conditioning for 24 hours, 10 and 28 days.

When testing materials with a known emission profile two samplings may be sufficient. If the concentration of VOCs in the test chamber is higher after 10 days than after 1 or 3 days, samples must be taken again after 28 $\pm$ 2 days.

The concentration of compounds in the test chamber air should reach the equilibrium before taking the first air sample.

The materials should be tested until the concentration of all emitted compounds (converted into the concentration in the standard room) is below half the threshold value for odour and irritation. After a test period of 56 days and with a minimum of 4 samplings, the concentration may be calculated by means of various mathematical models, e.g. first-order decay.

Note 1: For some materials air samples must be taken after 3, 10, 28, 56 or more days of conditioning.

Note 2: For long time testing only the determining compounds should be quantified at sample times later than 10 days. If the results, however, are used as documentation for other purposes, e.g. other labelling systems, it may be necessary to quantify all emitted compounds also after 28 days' conditioning.

Note 3: For long time testing the test specimen should be stored in a space at the same controlled indoor environmental conditions, if removed from the test chamber. The test specimen should be stored in the chamber at least 72 hours prior to air sampling.

9.4 Chemical Measurements

The chemical measurements include as a minimum identification and quantification of single VOC and aldehyde emissions from the test specimen.

An appropriate method for analysing single VOCs is sampling on Tenax followed by thermal desorption and detection by GC-MS.

Aldehydes may be sampled onto DNPH (2,4-dinitrophenylhydrazine) cartridges and analysed by high performance liquid chromatography (HPLC) and UV absorption.

At the same time, it is of great importance that a method capable of measuring emission of carcinogenic substances is used.

Note 1: Concentration of formaldehyde may also be determined by the spectro photometric method described in CEN prEN 717-1.

10 Sensory Evaluation

10.1 Overview of the Test Procedure

A sensory evaluation of the material emissions is used as a supplement to the chemical analyses. The sensory evaluation is carried out, as the human nose in many cases is superior to chemical analysis of the complex mixture of material emissions.

The air to be evaluated is extracted from a test chamber, which contains a test specimen of the material, and which is operated at given conditions.

A sensory panel evaluates the emissions of the material and the result determines in combination with the results of the chemical measurements the indoor-relevant time-value.

10.2 Sensory Panel

The sensory evaluation of the material emission is made by an untrained panel of at least 20 persons, who rate the air quality by marking the acceptability and odour intensity on continuous scales

The panel members should be in the age from 18 to 50 years. Panel members should have a normal olfactory sense. Smoking habits of each member should be noted. The sensory panel should preferably have an equal distribution of both sexes and not more than 40% smokers. The panellists should not suffer from a cold on the days of evaluation.

10.3 Panel Instructions

Prior to the evaluation of the air quality in the test chambers the members of the panel should be instructed to:

- refrain from eating garlic or spicy food on the day or the day before the evaluation
- refrain from eating or smoking during the last hour prior to evaluation
- have a high personal hygiene and refrain from using strong-smelling cosmetics
- wear clothes washed in a neutral detergent

Each panel member should be carefully instructed how to use the scales in the evaluation form. The scales used for recording acceptability and odour intensity of the air are shown in Appendix 1.

The panellists should take only one inhalation of the air to be evaluated and report their first impression of acceptability and odour intensity.

In case the panellists should evaluate the air quality from more than one test chamber, they should refresh their olfactory sense by breathing clean air for at least 1 minute in-between the evaluations. This is to avoid any cross adaptation.

10.3.1 Acceptability Scale

Acceptability of the perceived air quality should be marked on a continuous scale, slightly modified from Gunnarsen and Fanger (1992). The scale is divided into two separate scales with clearly marked end-points ”clearly acceptable”/”just acceptable” and ”just unacceptable”/”clearly unacceptable”, respectively.

The individual subject first decides whether the air quality is perceived as acceptable or unacceptable and then rate the degree of (un)acceptability. No marks are allowed between the labels ”just acceptable” and ”just unacceptable”.

10.3.2 Odour Intensity Scale

The perceived odour intensity of the air is evaluated by means of a separate scale. The scale has end-points and marks labelled as follows: ”no odour”, ”slight odour”, ”moderate odour”, ”strong odour”, ”very strong odour” and ”overwhelming odour”.

The acceptability and intensity of the supply air is evaluated by evaluation of the air from at least one empty test chamber.

11 Calculation of Acceptability and Odour Intensity

Calculations of acceptability and odour intensity are based on reading of the panellists' evaluations. The scales are assumed linear and reading is carried out by measuring the position of the marks on the scales.

To read the scales, they should be coded as follows:

11.1 Acceptability Scale

The end-point "clearly acceptable" is assigned the of +1, the end-point "clearly unacceptable" the value –1, while "just acceptable" and "just unacceptable" are assigned the values of +0.1 and –0.1, respectively.

11.2 Odour Intensity

Values are assigned to the labels as follows: 0 = "no odour", 1 = "slight odour", 2 = "moderate odour", 3 = "strong odour", 4 = "very strong odour", and 5 = "overwhelming odour".

At each time of evaluation the median of the panellists' evaluations of acceptability and odour intensity should be calculated for both the material sample and the empty test chamber.

The indoor air quality is regarded as acceptable at an acceptability greater than 0 and an odour intensity less than 2. Both criteria have to be fulfilled at the same time. The indoor-relevant time-value based on the sensory evaluations is given as the time (in days), at which the above-mentioned requirements for acceptability and odour intensity are fulfilled.

12 Calculation of the Indoor-Relevant Time-Value

The determination of the indoor-relevant time-value includes the following steps:

- Calculation of the specific emission rate
- Conversion of concentrations in the test chamber to concentrations in the standard room
- Determination of the indoor-relevant time-value based on the chemical analyses
- Determination of the indoor-relevant time-value based on the sensory evaluations

12.1 Calculation of the Specific Emission Rate

The specific emission rate, R_k, is calculated on basis of the measured emission test chamber concentrations, C_k, by the following Equation:

$$R_{k,i} = C_{k,i} \bullet n_k \bullet V_k/A_k = C_{k,i} \bullet n_k /L_k \ [\mu g/(m^2 h)] \qquad \text{(Eq. 2)}$$

where

$C_{k,i}$ = the measured test chamber concentration of i'th compound [$\mu g/m^3$]
n_k = air change rate in test chamber [1/h]
V_k = volume of test chamber [m^3]
A_k = exposed surface area of the test specimen [m^2]
L_k = loading factor [m^2/m^3]

12.2 Calculation of Concentrations in the Standard Room

The concentration of volatile organic compounds in the standard room is calculated on basis of the specific emission rates (Equation 2).

The emission rate in the standard room, R_s, is defined as:

$$R_{s,i} = C_{s,i} \cdot n_s \bullet V_s/A_s \ [\mu g/(m^2 h)] \qquad \text{(Eq. 3)}$$

where

$C_{s,i}$ = standard room concentration of the i'th compound [$\mu g/m^3$]
n_s = air change rate in the standard room [1/h]
V_s = volume of standard room [m^3]
A_s = exposed surface area of the product in the standard room [m^2]
L_s = loading factor [m^2/m^3]

The emission rates in the standard room and in the test chamber are assumed identical:

$$R_{s,i} = R_{k,i} \qquad \text{(Eq. 4)}$$

By combining Equations 2, 3, and 4, the concentration of the indoor air quality can be calculated by Equation 5:

$$C_{s,i} \bullet n_s \bullet V_s/A_s = C_{k,i} \bullet n_k \bullet V_k/A_k$$
$$\Downarrow$$
$$C_{s,i} = C_{k,i} \bullet n_k/n_s \bullet L_s/L_k \qquad \text{(Eq. 5)}$$

Conversion of the results from test chamber to indoor air quality is based on the standard room and standard conditions given in section 8.1.1.

When testing other products than those mentioned in section 8.1.1, the surface area specified in the product standard or the test and labelling criteria covering the specific product should be used.

12.3 Calculation of the Indoor-Relevant Time-Value

The indoor-relevant time-value states the time it takes to reach the acceptable concentration of VOCs in the standard room.

For information on the odour threshold (OT) and irritation threshold (IT) values, the database "VOCBase" [VOCBase, 1996] should be used.

The acceptable concentration of a single compound in the indoor air is 50% of the lowest value of OT and IT. The acceptable concentration is calculated using Equation 6:

$$C_{L,i} = 0.5 \bullet \min\{OT, IT\} \qquad \text{(Eq. 6)}$$

where

$C_{L,i}$ = the acceptable concentration of the i'th compound in the indoor air [$\mu g/m^3$]
OT = odour threshold value [$\mu g/m^3$]
IT = irritation threshold value [$\mu g/m^3$]

The irritative effect of more than one compound present at the same time is assumed bigger than that of a single compound. When more compounds are present at the same time, which is nearly always the case, the requirement is based on a sum of compounds, shown in Equation 7 below:

$$\sum_{i=1}^{n} \frac{C_{s,i}}{C_{L,i}} < 1 \qquad \text{(Eq. 7)}$$

Equation 7 does not apply for odorous compounds.

The indoor-relevant time-value is based on the highest time-value of both the chemical and sensory tests. The time-value is given in full days and rounded up to the nearest value, which can be divided by ten.

13 Test Report

An emission test report should as a minimum include the following:

Name and address of test laboratory
Unambiguous identification number of test report
Assignor's name and address
Purpose of the test

Description and identification of the product tested, including:
Trade name
Production date and number

Date of receipt at laboratory
Method of selection of test material
Conditioning of test material
Period of testing
Description of test method and procedures
Description of emission test chambers, including test conditions
Description of additional equipment
Deviations in procedure, conditions, etc. from the present standard
Measurements, evaluations and derived results, including:
Grounds of test method used
Test conditions, including time of measurements
Principle of determination of indoor-relevant time-values
Results in detail (allowing control of calculations)
Uncertainty of test results
Proposed declared indoor-relevant time-value

14 References

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Appendix 1

Example of scales used for sensory evaluation of acceptability and odour intensity.