BRITISH STANDARD

BS EN
12149 : 1998

Wallcoverings in roll form — Determination of migration of heavy metals and certain other elements, of vinyl chloride monomer and of formaldehyde release

The European Standard EN 12149 : 1997 has the status of a British Standard

ICS 91.180

National foreword

This British Standard reproduces verbatim EN 12149 : 1997 and implements it as the UK national standard.

The UK participation in its preparation was entrusted to Technical Committee CW/35, Wallcoverings, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled 'International Standards Correspondence Index', or by using the 'Find' facility of the BSI Standards Electronic Catalogue.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 12, an inside back cover and a back cover.

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Contents

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EUROPEAN STANDARD
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EN 12149

October 1997

ICS 91.180

Descriptors: Wall coverings, wall paper base, plastic coatings, tests, determination of content, metals, migrations, formaldehyde, vinyl chloride

English version

Wallcoverings in roll form — Determination of migration of heavy metals and certain other elements, of vinyl chloride monomer and of formaldehyde release

Revêtements muraux en rouleaux — Détermination de la migration de métaux lourds et certains autres éléments extractibles, de la teneur en chlorure de vinyle monomère et du dégagement de formaldéhyde

Wandbekleidungen in Rollen - Bestimmung der Migration von Schwermetallen und bestimmten anderen extrahierenden Elementen, des Gehaltes an Vinylchlorid Monomer sowie an Formaldehydabgabe

This European Standard was approved by CEN on 3 August 1997.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Ref. No. EN 12149 : 1997 E

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 99, Wallcoverings, of which the secretariat is held by BSI.

The following other European standards exist relating to wallcoverings:

EN 233 : 1989 (+A1 : 1996)	*Wallcoverings in roll form – Specification for finished wallpapers, wall vinyls and plastics wallcoverings*
EN 234 : 1989 (+A1 : 1996)	*Wallcoverings in roll form – Specification for wallcoverings for subsequent decoration*
EN 235 : 1989	*Wallcoverings in roll form – Vocabulary and symbols*
EN 259 : 1992 (+A1 : 1996)	*Wallcoverings in roll form – Specification for heavy duty wallcoverings*
EN 266 : 1992	*Textile wallcoverings*

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 1998, and conflicting national standards shall be withdrawn at the latest by April 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Contents

Introduction

This European standard has been prepared to cover factors which are considered to be environmental requirements of wallcoverings. The methods of test are based on those described in standards and published work as given in annex A.

1 Scope

This European standard specifies three methods of test concerning respectively:

– test A: the migration of heavy metals and certain other elements (antimony, arsenic, barium, cadmium, chromium, mercury, lead and selenium);

– test B: the determination of vinyl chloride monomer;

– test C: the determination of formaldehyde release.

This European Standard applies to all wallcoverings in roll form.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest editions of the publication referred to applies.

EN 235	*Wallcoverings in roll form – Vocabulary and symbols*
EN ISO 3696	*Water for analytical laboratory use – Specification and test methods* (ISO 3696:1987)
ISO 187	*Paper, board and pulps – Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

3 Definitions

For the purposes of this European standard, the definitions given in EN 235 apply.

4 Test A: Migration of heavy metals and certain other elements (antimony, arsenic, barium, cadmium, chromium, mercury, lead and selenium)

4.1 Principle

The soluble elements are extracted under conditions simulating the ingestion of the materials. The content present in the extract is then determined.

4.2 Reagents

4.2.1 During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and water of equivalent purity to grade 3 according to EN ISO 3696.

4.2.2 *Hydrochloric acid solution* (0,07 ± 0,005) mol/l.

4.2.3 *Hydrochloric acid solution* (2 ± 0,1) mol/l.

4.3 Test apparatus

4.3.1 Usual laboratory apparatus and glassware.

4.3.2 *pH meter*, having an accuracy of ± 0,2 pH units.

4.3.3 *Shaker*, capable of producing a linear reciprocating movement of an amplitude of (2 ± 0,5) cm with a frequency of 1 Hz.

4.3.4 *Oven*, capable of maintaining a constant temperature of (37 ± 2) °C.

4.3.5 *Filter*, with a pore size of 0,45 μm.

4.4 Test specimens

4.4.1 *Preparation of test specimens*

4.4.1.1 *Roll (except borders)*

Take a roll of wallcovering and make two cuts across the width of the wallcovering to produce a strip (30 ± 1) mm wide with a length the same as the width of the wallcovering. Repeat this at approximately 1 m intervals throughout the length of the roll. Cut the strips into rectangles (50 ± 1) mm long × (30 ± 1) mm wide (see figure 1).

4.4.1.2 *Borders*

Cut a strip of (30 ± 1) mm width through the whole length of the border. Cut this strip into rectangles of (50 ± 1) mm length. Use the appropriate number of rolls to produce at least 50 rectangles (see figure 2).

4.4.2 *Selection of test specimens*

Select by visual inspection the 10 rectangles which have the maximum amount of coating and/or the deepest yellow or red colour. Condition in accordance with ISO 187.

Cut these 10 rectangles into squares of approximately 6 mm × 6 mm and mix the squares obtained.

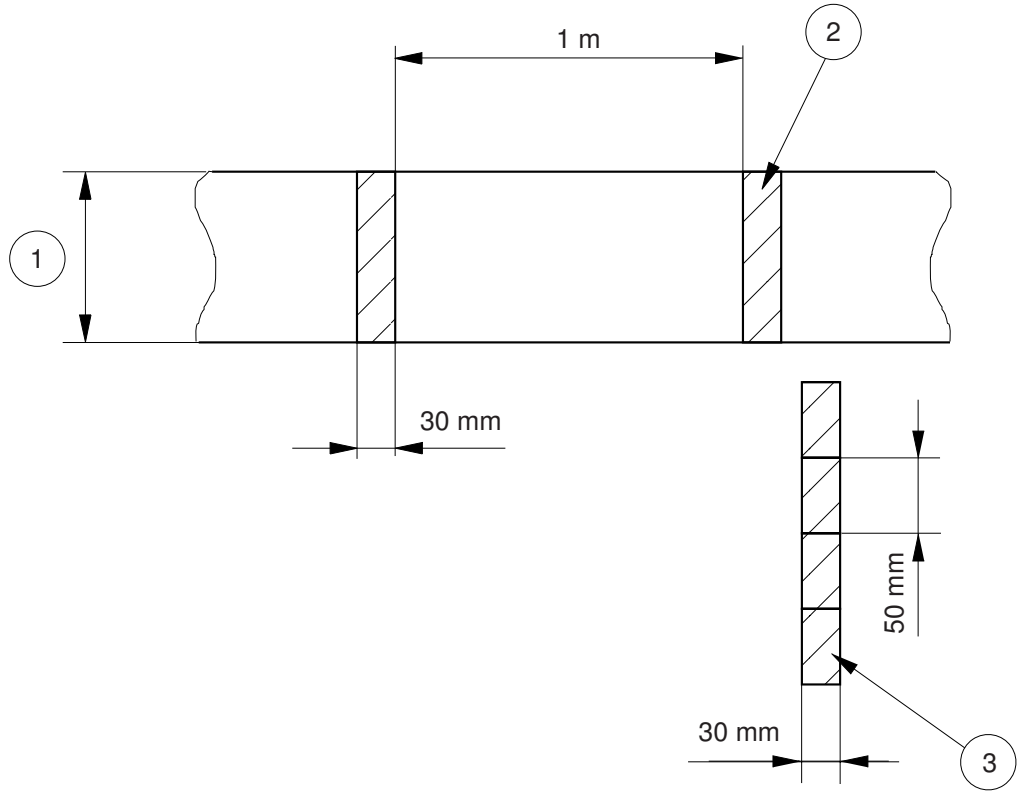
4.5 Procedure

4.5.1 *Extraction method*

Weigh to the nearest milligram (1 ± 0,05) g of the small squares (**4.4.2**) in a glass container of approximately 100 ml capacity. Add (50 ± 0,1) ml of the hydrochloric acid (**4.2.2**) at (37 ± 2) °C. Shake vigorously for 1 min, determine the pH of the solution (**4.3.2**).

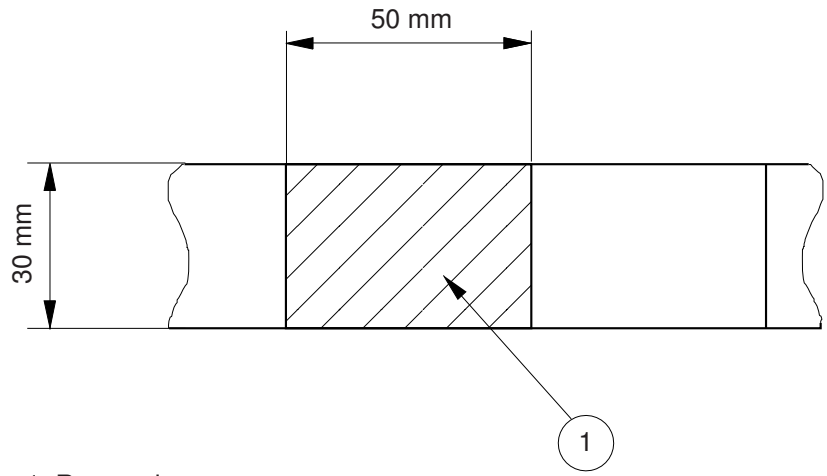
If the pH is higher than 1,5 add dropwise, while shaking, the hydrochloric acid (**4.2.3**) until the pH is below 1,5 and above 1,0.

Mount the container on a shaker (**4.3.3**) and place in the oven (**4.3.4**) at (37 ± 2) °C, shake the mixture at (37 ± 2) °C for (60 ± 2) min, then allow to stand at (37 ± 2) °C for (60 ± 2) min. Filter immediately through the 0,45 μm pore size filter (**4.3.5**) and collect the filtrate for the determination of the heavy metal (or other elements) content.



1. Roll width
2. Strip
3. Rectangles

Figure 1.



1. Rectangles

Figure 2.

4.5.2 *Determination of extracted heavy metals (and other elements)*

Use a method having a detection limit not exceeding 1/10 of the specified limit values.

NOTE 1. The detection limit is estimated at three times the standard deviation of the blank test value of the method being used.

NOTE 2. In principle, the methods given below (non-exhaustive list) satisfy the above condition:

– atomic absorption spectrometry;

– ICP emission spectrometry.

4.6 Expression of results

Calculate the heavy metal extracted for each metal (or element) in milligrams per kilogram of wallcovering from the following equation:

$$R = \frac{c}{m} \times 50 \qquad (1)$$

where

R is the determined heavy metal (or element) content, in milligrams per kilogram of wallcovering;

c is the concentration of the heavy metal (or element) in the extract, as determined by the method of **4.5.2**, in milligrams per litre;

m is the test portion, in grams.

5 Test B: Determination of vinyl chloride monomer

5.1 Principle

Dissolution or suspension of a test portion in a suitable solvent and determination of the vinyl chloride content by gas chromatography using the 'head-space' method.

5.2 Reagents

5.2.1 During the analysis, unless otherwise stated, use only reagents of recognized analytical grade suitable for gas chromatography and water of equivalent purity to grade 3 of EN ISO 3696.

5.2.2 *Vinyl chloride* (pure): lecture bottle fitted with fine adjustment valve.

5.2.3 *N,N-dimethyl acetamide.*

5.2.4 *Hydrogen.*

5.2.5 *Nitrogen.*

5.2.6 *Air.*

5.3 Standard solutions

5.3.1 *Preparation of stock solution*

Take 50 ml of N,N-dimethyl acetamide (**5.2.3**) and weigh. Bubble vinyl chloride through for approximately 10 s, until a concentration of approximately 10 g/l is obtained, re-weigh and seal the container.

5.3.2 *Diluted standard*

Take the solution prepared in accordance with **5.3.1** and further dilute by a factor of 500 (20 mg/l). For example, dilute 10 µl of stock to 5 ml with the N,N-dimethyl acetamide.

5.4 Test apparatus

5.4.1 Usual laboratory apparatus.

5.4.2 *Gas chromatograph*, fitted with an automatic headspace sampler, or with facilities for manual sample injection, and a flame ionization detector.

5.4.3 *Gas chromatographic column*, capable of completely separating the air peak, and the vinyl chloride peak from the standard solution (**5.3.2**) and the internal standard peak, if used.

The signal obtained from the column in use with a solution containing 0,02 mg of vinyl chloride per litre or kilogram shall be at least five times that of the background noise.

5.5 Test specimens

5.5.1 *Preparation of test specimens*

5.5.1.1 *Roll (except borders)*

Take a roll of wallcovering and make two cuts across the width of the wallcovering to produce a strip (30 ± 1) mm wide with a length the same as the width of the wallcovering. Repeat this at approximately 1 m intervals throughout the length of the roll. Cut the strips into rectangles (50 ± 1) mm long $\times$ (30 ± 1) mm wide (see figure 3).

5.5.1.2 *Borders*

Cut a strip of (30 ± 1) mm width through the whole length of the border. Cut this strip into rectangles of (50 ± 1) mm length. Use the appropriate number of rolls to produce at least 50 rectangles (see figure 4).

5.5.2 *Selection of test specimens*

Select by visual inspection the 10 rectangles most representative of the overall decoration. Condition in accordance with ISO 187.

Cut these 10 rectangles into squares of approximately 6 mm $\times$ 6 mm and mix the squares obtained.

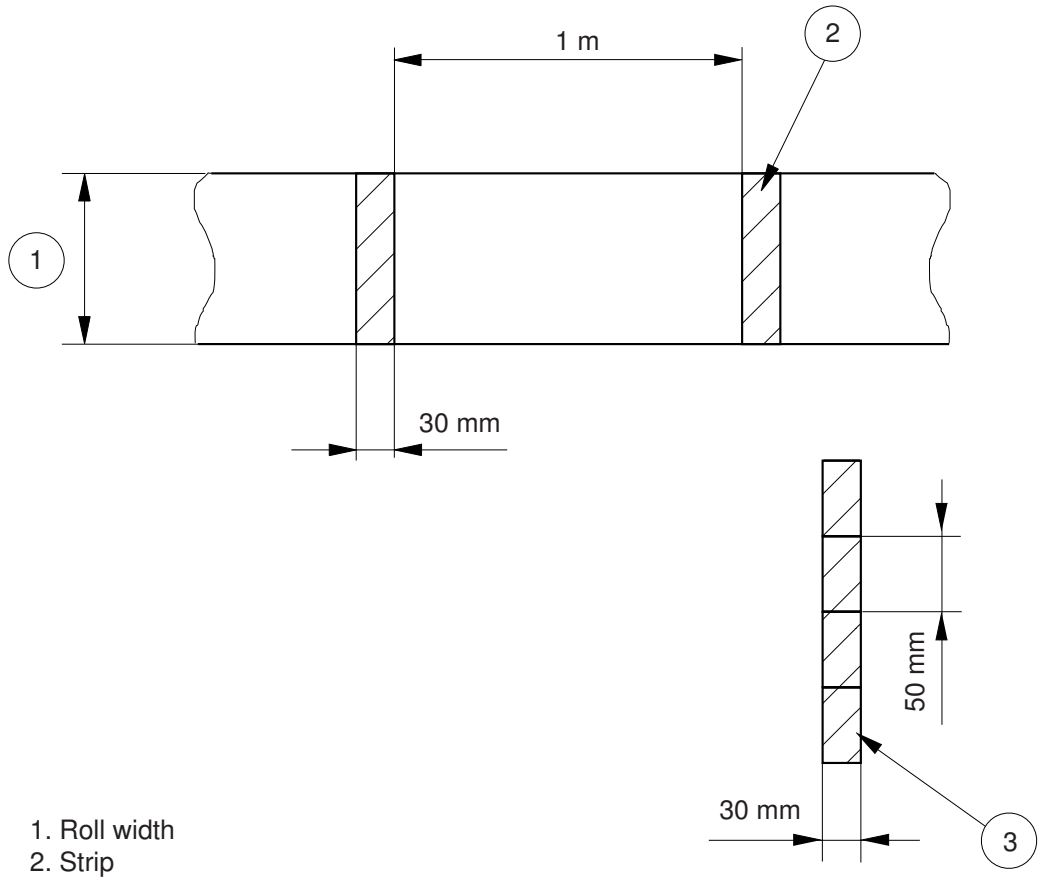
5.6 Procedure

5.6.1 *Preparation of test solutions*

Weigh accurately 0,5 g of the test specimens (**5.5.2**) into a headspace vial of 10 ml maximum capacity and add 2 ml of N,N-dimethyl acetamide (**5.2.3**). Close the vial.

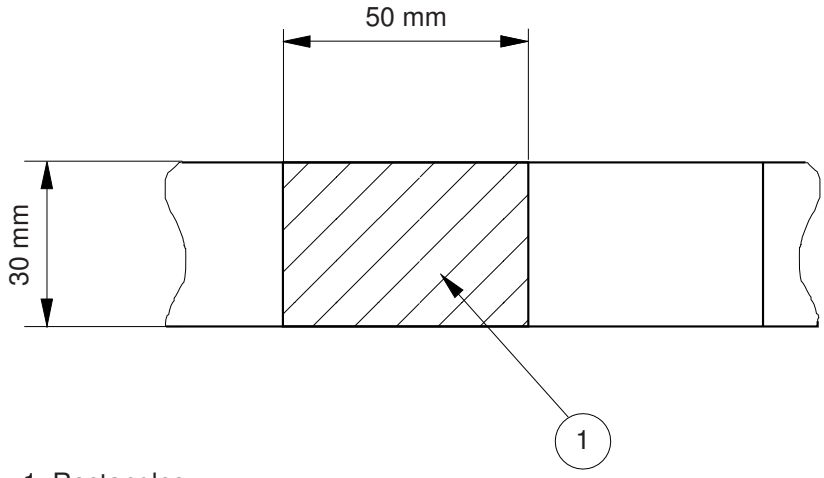
5.6.2 *Preparation of the calibration solution*

Into each of five vials, introduce 2 ml of N,N-dimethyl acetamide (**5.2.3**) and add to each the appropriate volume of the diluted standard solution such that a series of solutions is obtained approximately 0 ng, 125 ng, 250 ng, 375 ng and 500 ng of vinyl chloride monomer. Close the vials.



1. Roll width
2. Strip
3. Rectangles

Figure 3.



1. Rectangles

Figure 4.

5.6.3 ***Procedure for gas chromatography***

5.6.3.1 Place the vials containing the test solutions (**5.6.1**) and the calibration solutions (**5.6.2**) in a water bath controlled at (60 ± 1) °C for 2 h to attain equilibrium. Agitate the vials, avoiding contact between the liquid and stopper, to obtain, in the vials containing the test solutions, a suspension of resin as homogeneous as possible.

5.6.3.2 Take samples from the headspace of each vial.

If a manual sampling technique is used, take care to obtain a reproducible sample; in particular, warm the syringe to the same temperature as the sample.

5.6.3.3 Inject the samples (**5.6.3.2**) in turn, into the column and record the chromatograms.

5.7 Expression of results

Construct a calibration graph of vinyl chloride content of the calibration solution (**5.6.2**) against the corresponding peak areas.

Determine the vinyl chloride content of the test solutions (**5.6.1**) by interpolation from the calibration graph.

Calculate the vinyl chloride content W of the sample, in milligrams per kilogram, from the following equation:

$$W = \frac{CV}{m} \times 1000 \qquad (2)$$

where

C is the monomer vinyl chloride content, in milligrams per kilogram, of the test solution, determined from the calibration graph;

V is the volume, in millilitres, of solvent used;

m is the mass, in milligrams, of the test specimen.

6 Test C: Determination of formaldehyde using the modified WKl method

6.1 Principle

Formaldehyde release is determined by suspending test specimens over distilled water in a closed container at 40 °C during two periods of 24 h. The formaldehyde absorbed by the water during the second 24 h period is determined photometrically against a blank solution using acetylacetone as reagent.

6.2 Reagents

6.2.1 During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and water of equivalent purity to grade 3 of EN ISO 3696.

6.2.2 *Acetylacetone* (2,4-pentanedione > 99 %).

6.2.3 *Ammonium acetate.*

6.2.4 *Formaldehyde solution* (350 g/l to 400 g/l).

6.2.5 *Acetylacetone solution* (0,4 % *V/V*). Dilute 4 ml of the acetylacetone (**6.2.2**) to 1000 ml with water in a volumetric flask. Store in a sealed gas tight container and keep in the dark.

NOTE. Under these conditions this solution will remain stable for 4 weeks.

6.2.6 *Ammonium acetate solution* (200 g/l). Dissolve 200 g of the ammonium acetate (**6.2.3**) and make up to 1000 ml with water in a volumetric flask.

6.3 Standard solutions

6.3.1 The solutions in **6.3.2** to **6.3.5** shall be standardized before use.

6.3.2 *Iodine solution*, I_2 (0,05 mol/l).

6.3.3 *Sodium thiosulfate solution*, $Na_2S_2O_3$ (0,1 mol/l).

6.3.4 *Sodium hydroxide solution*, NaOH (1 mol/l).

6.3.5 *Sulfuric acid solution*, H_2SO_4 (1 mol/l).

6.3.6 *Starch solution*, 1 % (*m/m*).

6.4 Standard solution of formaldehyde

6.4.1 ***Solution A***

Dilute about 1 ml of the formaldehyde solution (**6.2.4**) to 1000 ml with water in a volumetric flask. Standardize the solution as follows.

Mix 20 ml of the solution A with 25 ml of the iodine solution (**6.3.2**) and 10 ml of the sodium hydroxide solution (**6.3.4**). After 15 min standing protected from light, add 15 ml of the sulfuric acid solution (**6.3.5**). Back-titrate the excess iodine with the sodium thiosulfate solution (**6.3.3**). Near the end of the titration, add some drops of the starch solution (**6.3.6**) as an indicator. Carry out a blank test with 20 ml of water (**6.2.1**) in parallel. Calculate the concentration of formaldehyde from the following equation:

$$C = (V_0 - V) \times C' \times \frac{1000}{20} \times 15 \qquad (3)$$

where

C is the formaldehyde concentration, in milligrams per litre;

V is the volume of sodium thiosulfate solution, in millilitres, for the test sample;

V_0 is the volume of sodium thiosulfate solution, in millilitres, for the blank;

C' is the concentration of standard sodium thiosulfate solution, in moles per litre.

6.4.2 ***Solution B***

Using the concentration determined in **6.4.1**, calculate the volume of solution A which will contain 15 mg of formaldehyde. Transfer this volume using a microburette to a 1000 ml volumetric flask and dilute to the mark with water.

NOTE. 1 ml of this solution contains 15 μg of formaldehyde.

6.5 Calibration standards

Dilute a range of calibration standards containing 0 μg to 15 μg of formaldehyde in 100 ml ground joint volumetric flasks as follows.

Table 1.

Volume of standard B ml	**Volume of water** ml	**Formaldehyde content** μg/ml
0	100	0
20	80	3
40	60	6
60	40	9
80	20	12
100	0	15

6.6 Apparatus

6.6.1 Usual laboratory apparatus.

6.6.2 *Volumetric flasks.*

6.6.3 *Burettes and microburettes.*

6.6.4 *Pipettes.*

6.6.5 *Oven.*

6.6.6 *Water bath*, capable of maintaining a temperature of (40 ± 2) °C.

6.6.7 *Spectrometer*, capable of measuring absorbance at a wavelength of 410 nm to 415 nm or fluorimeter capable of measuring a signal at 500 nm to 510 nm with quartz cells of 10 mm optical path length.

6.6.8 *Polyethylene or glass bottle*, of capacity 1000 ml, wide-necked with lid and equipped with a suitable hook device (see figure 7).

6.7 Test specimens

6.7.1 ***Preparation of test specimens***

6.7.1.1 *Roll (except borders)*

Take a roll of wallcovering and make two cuts across the width of the wallcovering to produce a strip (30 ± 1) mm wide with a length the same as the width of the wallcovering. Repeat this at approximately 1 m intervals throughout the length of the roll. Cut the strips into rectangles (50 ± 1) mm long × (30 ± 1) mm wide (see figure 5).

6.7.1.2 *Borders*

Cut a strip of (30 ± 1) mm width through the whole length of the border. Cut this strip into rectangles of (50 ± 1) mm length. Use the appropriate number of rolls to produce at least 50 rectangles (see figure 6).

6.7.2 ***Selection of test specimens***

Select fifty of the rectangles at random for the test. Condition in accordance with ISO 187 and weigh.

6.8 Procedure

6.8.1 Attach the fifty rectangles to a hook suspended from the lid of the 1000 ml bottle (see figure 7). Attach the strips so that the decorative face of the wallcoverings face each other and the samples do not touch the sides of the bottle or the liquid.

6.8.2 If the product is so thick that it is not possible to attach fifty pieces to the hook, attach the maximum possible number of rectangles, count and weigh them.

6.8.3 Add 50 ml of water to the 1000 ml bottle with a pipette of 50 ml capacity. Screw the lid gas tight and place the bottle in the oven at (40 ± 2) °C for 24 h.

6.8.4 After 24 h discard the liquid contained in the bottle and add a fresh 50 ml of water into the bottle.

6.8.5 Screw the lid with the strips gas tight and place the bottle in the oven at (40 ± 2)°C for a further 24 h.

6.8.6 Pipette 10 ml of the water from the bottle into a 50 ml ground joint bottle.

6.8.7 Pipette 10 ml of each formaldehyde calibration in separate 50 ml ground joint bottles.

6.8.8 Into each bottle add 10 ml of acetylacetone solution (**6.2.5**) and 10 ml of ammonium acetate solution (**6.2.6**). Stopper the flasks and shake them.

6.8.9 Warm the flasks for 15 min in the water bath maintained at (40 ± 2)°C. Remove the flasks from the water bath and allow to cool at room temperature for 1 h in the dark.

6.8.10 Measure the maximum absorbance at 410 nm to 415 nm or the fluorescence signal at 500 nm to 510 nm in 10 mm quartz cells against a water blank.

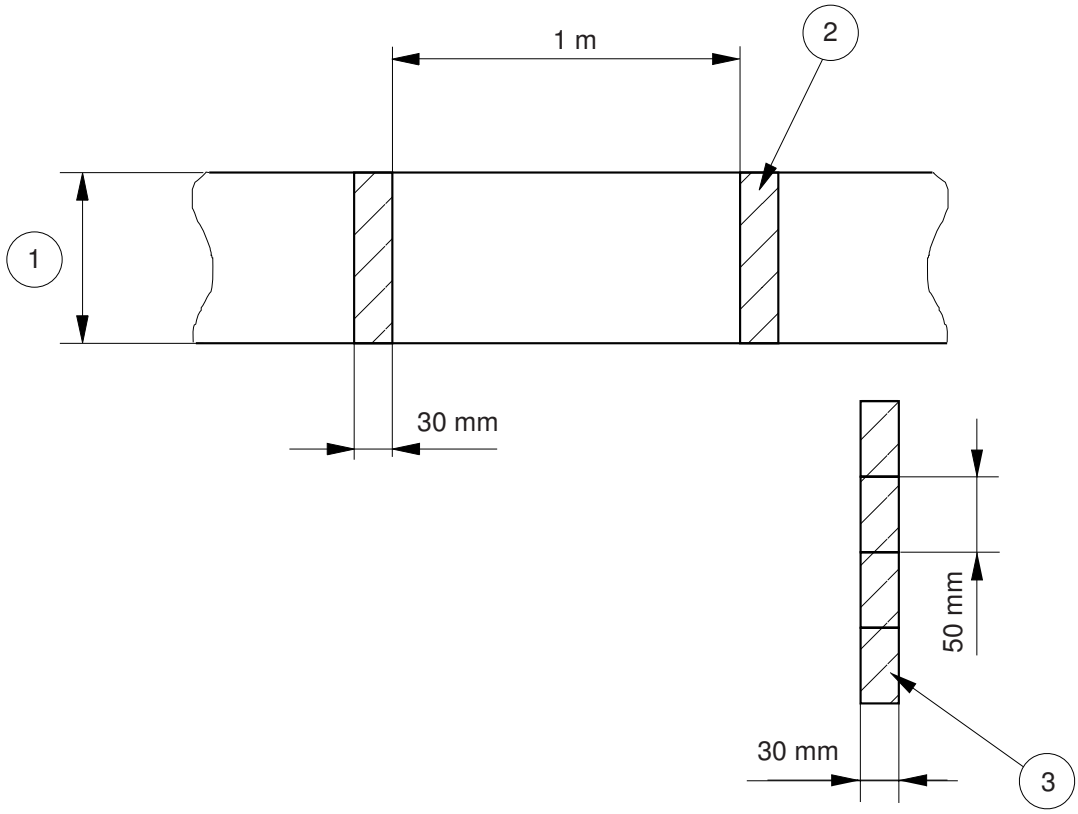
6.8.11 Carry out in parallel a blank test following all the same steps of the procedure.

6.9 Expression of results

Construct a calibration graph of concentration of formaldehyde against the absorbance or the fluorescence value.

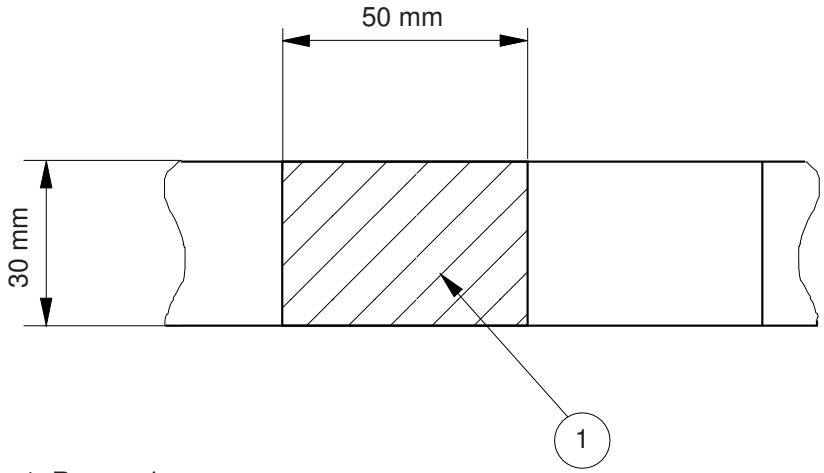
Read from the graph the amount of formaldehyde released by the sample according to its absorbance or fluorescence value.

Deduct the amount of formaldehyde in the blank from this result.



1. Roll width
2. Strip
3. Rectangles

Figure 5.



1. Rectangles

Figure 6.

Calculate the amount of formaldehyde released from wallcovering, expressed in milligrams per kilogram of wallcovering, from the following equation:

$$G = \frac{50A}{m} \quad (4)$$

where

m is the mass of pieces attached to the hook, in grams;

G is the amount of formaldehyde released from the wallcovering, in milligrams per kilogram;

A is the result of spectrometric measurement corrected from the blank in micrograms per millilitre.

Calculate the amount of formaldehyde released from wallcovering, expressed in milligrams per square metre, from the following equation:

$$F = \frac{100A}{3n} \quad (5)$$

where

A is the result of spectrometric measurement corrected from the blank, in micrograms per millilitre;

n is the number of pieces 30 mm $\times$ 50 mm attached to the hook;

F is the amount of formaldehyde released, in milligrams per square metre.

7 Test report

The test report shall contain the following information:

a) a reference to this European Standard, EN 12149:1997;

b) a reference to the test carried out, test A, B or C;

c) a complete identification of the product tested, e.g., pattern number, batch number;

d) for test A, the method used for the determination of each metal (or element);

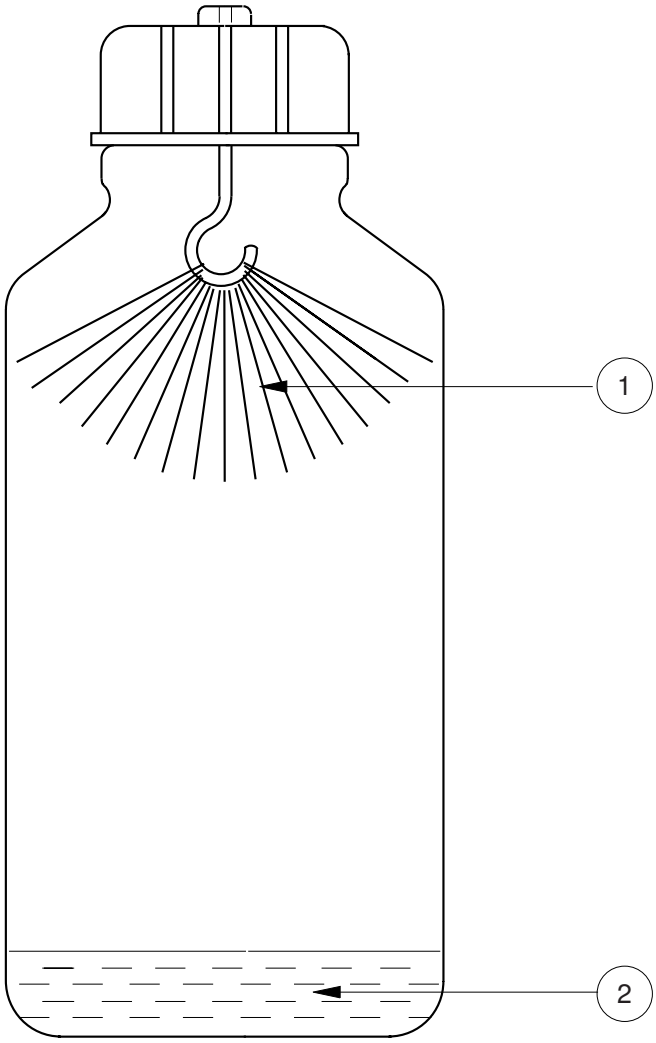
e) the test results according to clause:

4.6 for test A;

5.7 for test B;

6.9 for test C;

f) any deviation from this standard that may have affected the results.



1. 50 wallcovering strips
2. 50 ml of water

Figure 7.

Annex A (informative)
Bibliography

EN 71-3:1995	*Safety of toys – Migration of certain elements*
EN 717-3	*Wood-based panels – Determination of formaldehyde release – Formaldehyde release by the flask method*
ISO 6401	*Plastics – Homopolymer and copolymer resins of vinyl chloride – Determination of residual vinyl chloride monomer – Gas chromatographic method*
Roffael 1975	*The Wilhelm-Klauditz Institute flask method*

blank

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