

BS EN 14602:2012



BSI Standards Publication

Footwear — Test methods for the assessment of ecological criteria

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National foreword

This British Standard is the UK implementation of EN 14602:2012. It supersedes BS EN 14602:2004 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee TCI/69, Footwear, leather and coated fabrics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

Footwear - Test methods for the assessment of ecological criteria

Chaussure - Méthodes d'essai pour l'évaluation de critères
écologiquesSchuhe - Prüfverfahren zur Beurteilung ökologischer
Kriterien

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Foreword

This document (EN 14602:2012) has been prepared by Technical Committee CEN/TC 309 "Footwear", the secretariat of which is held by AENOR.

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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 14602:2004.

The main technical changes that have been made in this new edition of EN 14602 are the following ones:

- changes in the general organisation of the standard; all the criteria for the Ecolabel for footwear that are already covered by European standards are removed from this edition;
- improvement in the test methods for the determination of total heavy metals content;
- criteria on COD and Cr III have been introduced;
- new reference test method for the determination of phthalates;
- new test method for the determination of the electrical energy consumption.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard defines certain test methods necessary to issue the footwear Ecolabel. For some criteria, this European Standard provides important clarification or gives a test method to assess the ecological criteria.

NOTE The footwear Ecolabel has been published in the Official Journal of July 28th, 2009.

This European Standard applies to any kind of footwear except those containing electrical or electronic components.

The chemical analysis of the metallic components is outside of the scope of this European Standard.

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.

EN 1122:2001, *Plastics — Determination of cadmium — Wet decomposition method*

EN 12868, *Child use and care articles — Methods for determining the release of N-Nitrosamines and N-Nitrosatable substances from elastomers or rubber teats and soothers*

EN ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885)*

CEN ISO/TS 16181, *Footwear — Critical substances potentially present in footwear and footwear components — Determination of phthalates in footwear materials (ISO/TS 16181)*

EN ISO 17072-2:2011, *Leather — Chemical determination of metal content — Part 2: Total metal content (ISO 17072-2:2011)*

EN ISO 17294-2, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of 62 elements (ISO 17294-2)*

ISO 8288, *Water quality — Determination of cobalt, nickel, copper, zinc, cadmium and lead — Flame atomic absorption spectrometric methods*

3 Terms and definitions

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

3.1
volatile organic compound
VOC
organic compound that has, at 293,15 K, a vapour pressure of 0,01 KPa or more, or that has a corresponding volatility under the particular conditions of use

3.2
volatile organic compounds emissions
VOC emissions
amount of volatile organic compounds emitted to the atmosphere to produce a pair of shoes

3.3

test period

T_p

consecutive test period during which:

- the production of the shoe or a group of shoes being analysed is well known;
- the consumption of chemical preparations is well known

3.4

process electric consumption

PEC

electricity used only by the process equipment used to manufacture the footwear

4 Test methods associated to the criteria

NOTE The uncertainty of measurement for each test method described in this European Standard can be assessed according to the methods described ENV 13005 or ISO 5725-2.

4.1 Determination of total heavy metals content

4.1.1 General

The total concentration of heavy metals in a product is considered as a relevant criterion regarding the end of life disposal. This test method identifies the main chemicals of potential hazard which may contaminate the environment after disposal or incineration.

There shall be no Arsenic, Cadmium and Lead in the materials used for the product assembly or in the final product. Presence of heavy metals shall be established by either:

- testing the materials individually; or
- testing the upper and lower components of the shoe as composite groups of materials.

NOTE The total concentration of other heavy metals (see Table 1) can be determined using this method.

Table 1 — Possible heavy metals determined

Aluminium (Al)	Mercury (Hg)
Antimony (Sb)	Molybdenum (Mo)
Barium (Ba)	Nickel (Ni)
Boron (B)	Potassium (K)
Chromium (Cr)	Selenium (Se)
Cobalt (Co)	Silicon (Si)
Copper (Cu)	Tin (Sn)
Iron (Fe)	Titanium (Ti)
Magnesium (Mg)	Zinc (Zn)
Manganese (Mn)	Zirconium (Zr)

4.1.2 Preparation of the samples

4.1.2.1 Testing the materials for the product assembly

Test each material individually.

Each material is ground according to EN ISO 4044 or a similar test method.

NOTE Textile materials do not need to be ground.

4.1.2.2 Testing the final product

Separate the upper and the lower components of the footwear.

Fully grind each group of components separately according to EN ISO 4044 or a similar test method.

NOTE Only a full grinding of the test specimen will ensure the reproducibility of the test results.

4.1.3 Reagents

WARNING — Violent reactions may be produced with perchloric acid (HClO_4) on an organic matrix.

4.1.3.1 H_2SO_4 , 96 % (mass fraction).

4.1.3.2 HNO_3 , 65 % (mass fraction).

4.1.3.3 HClO_4 , 65 % (mass fraction).

4.1.3.4 HCl , 36 % (mass fraction).

4.1.3.5 Demineralised water.

4.1.4 Apparatus and materials

Standard laboratory apparatus and glassware, together with the following:

4.1.4.1 A suitable heating device (for the acid digestion only).

4.1.4.2 Volumetric flask, of 100 ml.

4.1.4.3 Filter paper of 0,45 μm mesh size.

4.1.4.4 Microwave reactor (for the microwave digestion only), capable of working at maximum 100 bars (or 1 450 psi).

4.1.5 Digestion

4.1.5.1 Method A – acid digestion

Prepare, according to 4.1.2, a test piece of $(0,500 \pm 0,005)$ g of ground sample. The digestion given in EN 1122:2001, 6.3.2, method B is to be used.

4.1.5.2 Method B – acid digestion

Prepare, according to 4.1.2, a test piece of $(1,000 \pm 0,005)$ g of ground sample. The digestion given in EN ISO 17072-2:2011, 6.5.1 is to be used.

4.1.5.3 Method C - Microwave digestion

Prepare, according to 4.1.2, a test piece of 0,100 g to 0,500 g of ground sample.

Prepare a digestion solution by mixing 5 ml of demineralised water (4.1.3.5) and 5 ml of the following mixture: HNO₃, 65 % (4.1.3.2) and HCl, 36 % (4.1.3.4) (1/3:2/3).

Put the test piece in the microwave reactor (4.1.4.4), add the digestion solution and then start the digestion cycles (see for example a programme in Table 2).

Table 2 — Cycles of digestion

Time (min)	2,5	2,5	5	10	5	2,5	2,5	5	5
Power (W)	250	0	350	500	0	250	0	350	550

When the cycles are completed, transfer the resulting solution, filtering it through a filter paper (4.1.4.3), to a volumetric flask (4.1.4.2) and adjust the volume to 100 ml with demineralised water (4.1.3.5).

One blank (acid solution) is treated at the same time and a validation with this reference solution is carried out.

The digestion vessels shall be cleaned with special attention once the acid digestion is finished.

4.1.6 Determination of heavy metals content

Table 3 defines the recommended options for the determination of heavy metals in footwear.

Table 3 — Test method for heavy metals determination

	Testing the materials		Testing the final product	
	Soft material leather, textile	Hard material polymers	Upper	Lower components of the footwear
Method A	possible	Recommended (especially for PVC)	possible	Recommended (especially for PVC)
Method B	possible	possible	possible	possible
Method C	possible	possible	possible	possible

From the solution obtained in 4.1.5.1 or 4.1.5.2 or 4.1.5.3 determine, within 24 h, the Cd, As, Pb content by atomic absorption spectroscopy (AAS) as specified in ISO 8288, or by inductively coupled plasma atomic emission spectroscopy (ICP) as specified EN ISO 11885 or EN ISO 17294-2 (see EN ISO 17072-2).

NOTE 1 The quantification limit for a 1 g sample is given in Table 4.

Table 4 — Test method for heavy metals determination

Metal	Limit of quantification in the liquid extraction	Limit of quantification
	µg/l	mg/kg
As	20	5
Cd	5	1
Pb	20	5

Results shall be expressed as:

- in mg/kg of material for each metal (Cd, Pb, and As) for the procedure in 4.1.2.1;
- in mg/kg of footwear sample for each metal (Cd, Pb, and As) for the procedure in 4.1.2.2.

NOTE 2 Other metals (see Table 1) can be determined by this method.

Using the acid digestion (method B), if Pb is detected, the digestion procedure shall be repeated but replacing H₂SO₄ (4.1.3.1) by HCl (4.1.3.4).

4.1.7 Test report

The test report shall include at least the following information:

- a) reference to this method, including digestion procedure and determination method;
- b) complete description of the sample, including the material type and all the details concerning the mass of metallic and non metallic materials;
- c) heavy metals content according to Table 4,
 - 1) in mg/kg of material, for each metal Cd, Pb, As, as calculated in 4.1.6 (procedure 4.1.2.1);
 - 2) in mg/kg of footwear, for each metal Cd, Pb, As, as calculated in 4.1.6 (procedure 4.1.2.2);
- d) date of testing;
- e) for samples containing leather, a determination of dry matter shall be reported as required by EN ISO 17072-2;
- f) any deviation from this test method.

4.2 Water consumption, emission in water of COD and chromium III

If the footwear contains leather, it is the responsibility of the footwear manufacturer to require the water consumption and emission in water of COD and chromium III for the tanning.

These information are given by the tanner.

Leather is produced after several operations (beamhouse, tanning, dyeing, finishing...) that are not always done at the same location. The consumption figures for hides and skins shall include all the different steps. To collect all this information with a guarantee of tracability can be difficult.

NOTE In Europe, the information concerning COD (chemical oxygen demand) and chromium III in water is available in all the industry due to the application of Directive 91/271/EEC and the appropriate national regulations.

4.3 Determination of nitrosamines

Separate the rubber components from the footwear, grind them separately according to 4.1.2 and determine the nitrosamines content according to the test method given in EN 12868.

For this test method, a detection limit of 0,1 mg/kg for rubber has been determined. Any nitrosamine content below the detection limit shall be reported as “none detected”.

4.4 Determination of phthalates

Separate the PVC components from the footwear, grind them separately according to 4.1.2 and determine the phthalates content (see the list of the phthalates Table 5) according to the test method given in CEN ISO/TS 16181.

Table 5 — Phthalates to be determined

Phthalates	Symbol
(bis(2-ethylhexyl)phthalate)	DEHP
butylbenzylphthalate	BBP
dibutylphthalate	DBP
di-isononyl phthalate	DINP
di-isodecyl phthalate	DIDP
di-n-octyl phthalate	DNOP

4.5 Determination of the VOC emissions

4.5.1 General

VOC emissions from purchased leather, adhesives, finishes and production of footwear shall be calculated for a period of at least six months.

NOTE This calculation can be misleading for small amounts of production.

4.5.2 Procedure

The total amount of VOCs used in the shoe production shall be calculated as follows:

$$M_{\text{VOCtotal}} = \sum (M_{\text{adhesives}} \times C_{\text{VOCa}}) + \sum (A_{\text{finishes}} \times M_{\text{finishes}} \times C_{\text{VOCf}})$$

where

M_{VOCtotal} is the total amount of VOCs used in the production of the pair of shoes, in g;

$M_{\text{adhesives}}$ is the amount of adhesives applied to the pair of shoes considered, in g; only adhesives with solvents have to be taken into account; water based and hot melt adhesives are exempted;

C_{VOCa} is the VOC content of the adhesives applied, in g of VOCs per g of adhesive;

A_{finishes} is the area of the pair of shoes onto which the finish¹⁾ is applied, in m²;

M_{finishes} is the amount of finish applied per metre square, in g/m²;

C_{VOCf} is the VOC content of the finishes applied, in g of VOCs per g of finish.

4.5.3 Test report

The test report shall include at least the following information:

- a) reference to this test method;
- b) complete description of the type of shoe being analysed;
- c) average mass of the representative pair of shoes to which the results apply and how it has been determined;
- d) description of the process steps taken into account in the calculation;
- e) the test period: for example, "3 months: November 2010 to January 2011";
- f) result obtained, M_{VOCtotal} , in 4.5.2, in g of VOC per pair of shoes;
- g) date of testing;
- h) any deviation from this test method.

4.6 Determination of the electrical energy consumption

The applicant shall declare the energy consumption referred to the assembly final process of the footwear. The Average Energy Consumption AEC shall be calculated:

On the basis of the overall production of shoes of the plant using the formula:

$$\text{AEC} = \frac{\text{MJ}_{\text{dp}}}{N} \quad (1)$$

where

MJ_{dp} = average energy used per day in production of shoes, including electrical power and fossil fuel, calculated on annual basis;

N = average number of pair of shoes produced per day, calculated on annual basis.

On the basis of the production of eco-labelled shoes of the plant using the formula:

$$\text{AEC} = \frac{\text{MJ}_{\text{ep}}}{N} \quad (2)$$

where

MJ_{ep} = average energy used per day in production of shoes, including electrical power and fossil fuel, calculated on annual basis;

1) Finishes: base coats, top coats and repair coats, (upper) finish layers of leather, synthetics upper, lining, cotton, etc. only when based on solvents.

N = average number of pair of shoes produced per day, calculated on annual basis.

Conversion of energy power expression: in order to standardise the energy expression unit, the electrical energy and the fossil fuel consumption shall be expressed in MJ value. In order to calculate the MJ value, the following relation shall be considered:

— Electrical energy: 1 Kw/h = 3,6 MJ [11]

— Fossil fuel: The fossil fuel energy content shall be calculated on the basis of the following table of conversion for the main products used in energy production.

Table 6 — Higher Heating Value (HHV) of some common fuels [12]

Fuel	HHV MJ/kg	Fuel	HHV MJ/kg
Methane	55,50	Coal (Anthracite)	27,00
Propane	50,35	Coal (Lignite)	15,00
Gasoline	47,30	Wood	15,00
Kerosene	46,20	Peat (damp)	6,00
Diesel	44,80	Peat (dry)	15,00

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