

BRITISH STANDARD

**BS EN
12748:1999**

Footwear — Test methods for outsoles, insoles, linings and insocks — Water soluble content

The European Standard EN 12748:1999 has the status of a
British Standard

ICS 61.060

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National foreword

This British Standard is the English language version of EN 12748:1999. It supersedes method 5 of BS 1309:1974, which is deleted by amendment.

The UK participation in its preparation was entrusted to Technical Committee TCI/69, Footwear and leather, 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;
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English version

Footwear — Test methods for outsoles, insoles, lining and insocks
— Water soluble content

Chaussures - Méthodes d'essai applicables aux premières de montage, aux doublures, aux premières de propreté et aux semelles d'usure - Détermination des substances solubles dans l'eau

Schuhe - Prüfverfahren für Laufsohlen, Brandsohlen, Futter und Decksohlen - Wasserlöslicher Anteil

This European Standard was approved by CEN on 9 August 1999.

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Foreword

This European Standard 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of an identical text or by endorsement, at the latest by March 2000, and conflicting national standards shall be withdrawn at the latest by March 2000.

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.

1 Scope

This standard specifies a method for the determination of the water soluble contents for outsoles, insoles, lining and insocks.

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 edition of the publication referred to applies.

EN 12222 *Footwear - Standard atmospheres for conditioning and testing of footwear and components for footwear.*

prEN 13400:1998 *Footwear. Sampling location of components for footwear.*

3 Definitions

For the purpose of this standard the following definitions apply:

water soluble matter

the quantity of all those substances which under certain conditions are dissolved out of the material by water.

water soluble inorganic substances

the sulfates ash of water soluble substances.

water soluble organic substances

the difference between total water solubles and sulfated ash of water solubles.

4 Apparatus and material

The following apparatus and material shall be used:

4.1 650 ml to 750 ml flask with a wide neck and close-fitting glass or rubber stopper.

4.2 Fluted filter (185 mm diameter).

4.3 500 ml measuring vessel.

4.4 50 ml delivery pipette.

4.5 Quartz, platinum or porcelain evaporating basin with flat bottom, to hold 50 ml, and suitable desiccators.

4.6 Funnel and 300 ml Erlenmeyer flask.

4.7 Distilled water.

4.8 Appropriate shaker apparatus with (50 ± 10) revolutions per minute.

4.9 Thermometer.

4.10 Laboratory balance with a sensitivity of 0.1 mg.

4.11 Analytical balance.

4.12 Suitable oven set to $102\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

4.13 Water bath.

4.14 1 mol/l sulfuric acid.

4.15 Muffle oven at $800\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$.

5 Sampling

Test specimens shall be taken in accordance with prEN 13400:1998.

The material shall be ground and extracted with dichloromethane using a soxhlet apparatus for a minimum of 30 refluxes of solvent. Condition the material for 24 hours in accordance with EN 12222. Minimum two test pieces are necessary.

6 Test method

6.1 Shaking in water

Shake mechanically at (50 ± 10) revolutions per minute for 2 h, 10 g conditioned ground and dichloromethane extracted material with 500 ml distilled water at $23\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ in a wide-necked flask.

6.2 Filtrate

Filter the contents of the flask through a fluted filter until clear. Discard the first 50 ml of the filtrate. Determine the soluble organic and inorganic substances in a further 50 ml of the subsequent filtrate.

6.3 Total water solubles

Evaporate on the water bath until dry, exactly 50 ml of the filtrate in a previously weighed dish heated at $800\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$, drying at $102\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for approx. 2 h; cool in the desiccator; weigh quickly. Only one dish at a time shall be put into a small desiccator and at most two in a large desiccator. Repeat drying until the reduction in mass amounts to less than 2 mg, but not for more than 8 h.

6.4 Sulfated ash of water solubles

Thoroughly wet the residue obtained in accordance with 6.3 in the dish with a few drops of 1 mol/l sulfuric acid, fume over a low flame until no sulfuric acid vapour is visible. Heat until red hot, preferably in a muffle oven, at $800\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ for 15 min. Cool in the desiccator and weigh as quickly as possible. Repeat the addition of acid, heating, cooling and weighing until the mass of the residue is constant.

NOTE: If the mass of water soluble inorganic matter is likely to be less than 2,0 %, it is recommended that 100 ml or 200 ml aliquot portion should be used.

7 Expression of results

7.1 The total water solubles, m_{ws} , in per cent, is given by the equation:

$$m_{ws} = \frac{r_d \times 10 \times 100}{m_c}$$

where

r_d , is the mass of dry residue, in grams;

m_c , is the original mass of the component, in grams.

7.2 Sulfated ash of water solubles, m_{saws} , in per cent, is given by the equation

$$m_{\text{saws}} = \frac{r_{\text{si}} \times 10 \times 100}{m_{\text{c}}}$$

where

r_{si} is the mass of sulfated residue from ignition, in grams.

7.3 Water soluble organic substances are the difference between the total water soluble substances and the water soluble inorganic substances.

The result is the average of the two values obtained for each test piece.

8 Test Report

The test report shall include the following information:

- a) results, expressed in accordance with clause 7, rounded up or down to 1 decimal place;
- b) full identification of the sample;
- c) reference to this method of test;
- d) date of testing.

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BRITISH STANDARD

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12749:1999**

Footwear — Ageing conditioning

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