BRITISH STANDARD

BS EN
1744-4:2005

Tests for chemical properties of aggregates —

Part 4: Determination of susceptibility of fillers for bituminous mixtures

The European Standard EN 1744-4:2005 has the status of a British Standard

ICS 91.100.15

BSI
British Standards

National foreword

This British Standard is the official English language version of EN 1744-4:2005.

The UK participation in its preparation was entrusted by Technical Committee B/502, Aggregates, to Subcommittee B/502/6, Test methods, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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

Cross-references

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 15 and a back cover.

The BSI copyright notice displayed in this document indicates when the document was last issued.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 29 September 2005

ISBN 0 580 46618 3

Amendments issued since publication

Amd. No.	Date	Comments

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 1744-4

August 2005

ICS 91.100.15

English version

Tests for chemical properties of aggregates - Part 4: Determination of water susceptibility of fillers for bituminous mixtures

Essais pour déterminer les propriétés chimiques des granulats - Partie 4 : Détermination de la sensibilité à l'eau des fillers pour mélanges bitumeux

Prüfverfahren für chemische Eigenschaften von Gesteinskörnungen - Teil 4: Bestimmung der Wasserempfindlichkeit von Füllern in bitumenhaltigen Mischungen

This European Standard was approved by CEN on 27 June 2005.

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, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 1744-4:2005: E

Contents

page

Foreword

This European Standard (EN 1744-4:2005) has been prepared by Technical Committee CEN/TC 154 “Aggregates”, the secretariat of which is held by BSI.

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 2006, and conflicting national standards shall be withdrawn at the latest by February 2006.

This European Standard forms part of a series of tests for chemical properties of aggregates. Test methods for other properties of aggregates are covered by Parts of the following European Standards:

EN 932 Tests for general properties of aggregates

EN 933 Tests for geometrical properties of aggregates

EN 1097 Tests for mechanical and physical properties of aggregates

EN 1367 Tests for thermal and weathering properties of aggregates

EN 13179 Tests for filler aggregate used in bituminous mixtures

The other parts of EN 1744 are, or will be:

Part 1: Chemical analysis

Part 2: Determination of resistance to alkali/aggregate reaction

Part 3: Preparation of eluates by leaching of aggregates

Part 5: Determination of acid soluble chloride salts

Part 6: Determination of the influence of aggregate extract on the initial setting time of cement

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

1 Scope

This European Standard specifies the procedure for the determination of the water susceptibility of fillers for bituminous mixtures, by separation of filler from a bitumen filler mixture.

A method for the determination of water susceptibility by volume increase and loss of stability of a Marshall specimen is described in Annex A.

2 Normative references

The following referenced documents are indispensable for the application of this European Standard. For dated references, only the edition cited applies. For undated references the latest edition of the referenced document (including any amendments) applies.

EN 932-1, *Tests for general properties of aggregates — Part 1: Methods for sampling*

EN 932-2, *Tests for general properties of aggregates — Part 2: Methods for reducing laboratory samples*

EN 932-5, *Tests for general properties of aggregates — Part 5: Common equipment and calibration*

EN 933-2, *Tests for geometrical properties of aggregates — Part 2: Determination of particle size distribution -Test sieves, nominal size of apertures*

EN 933-3*, Tests for geometrical properties of aggregates - Part 3: Determination of particle shape - Flakiness index*

EN 933-4*, Tests for geometrical properties of aggregates - Part 4: Determination of particle shape - Shape index*

EN 12697-6, *Bituminous mixtures — Test methods for hot mix asphalt — Part 6: Determination of bulk density of bituminous specimens*

EN 12697-12, *Bituminous mixtures — Test methods for hot mix asphalt — Part 12: Determination of the water sensitivity of bituminous specimens*

EN 12697-30, *Bituminous mixtures - Test methods for hot mix asphalt - Part 30: Specimen preparation by impact compactor*

EN 12697-34, *Bituminous mixtures — Test methods for hot mix asphalt — Part 34: Marshall test*

EN 12697-35, *Bituminous mixtures — Test methods for hot mix asphalt — Part 35: Laboratory mixing*

EN 13357, *Bitumen and bituminous binders — Determination of the efflux time of petroleum cut-back and fluxed bitumens*

3 Terms and definitions

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

3.1
filler aggregate
aggregate, most of which passes a 0,063 mm sieve, which can be added to construction materials to provide certain properties

3.2
water susceptibility of filler
measure of the degree of separation which occurs in the presence of water from a filler bitumen mixture, e.g. as a result of intra-crystalline water inclusion between aggregate particles and binder coating

3.3
subsample
sample obtained from sampling increments or a bulk sample by means of a sample reduction procedure

3.4
test portion
sample used as a whole in a single test

3.5
aggregate size
designation of aggregate in terms of lower *(d)* and upper *(D)* sieve sizes expressed as *d/D*

NOTE This designation accepts the presence of some particles which will be retained on the upper sieve (oversize) and some which will pass the lower sieve (undersize).

3.6
particle size fraction
fraction of an aggregate passing the larger of two sieves and retained on the smaller

NOTE The smaller sieve size can be zero.

3.7
constant mass
successive weighings after drying at least 1 h apart not differing by more than 0,1 %, by mass

NOTE In many cases constant mass can be achieved after a test portion has been dried for a pre-determined period in a specified oven at (110 ± 5) °C. Test laboratories can determine the time required to achieve constant mass for specific types and sizes of sample dependent upon the drying capacity of the oven used.

4 Principle

A mixture of filler and bitumen is stirred in hot water. If filler becomes separated from the mixture (indicated by the turbidity of the water), the filler is recovered on a filter paper and weighed.

5 Separation of filler from a bitumen filler mixture

5.1 Reagents

5.1.1 *Bitumen*:50/70.

5.1.2 *Redistilled Kerosene* (paraffin oil), petroleum distillate with a boiling range between 190 °C and 260 °C.

NOTE The displacement liquid used in the method of testing density of cement, as specified in EN 196-6, is suitable.

5.1.3 *Low viscosity bitumen solution,* obtained by dissolution of 50/70 bitumen (5.1.1) in kerosene (5.1.2), with viscosity at 25 °C of (240 ± 10) (St) ((60s ± 5) s S.T.V. (Standard Tar Viscometer) 10 mm) as specified in EN 13357.

5.1.4 *Demineralized water.*

5.2 Apparatus

5.2.1 *All apparatus*, unless otherwise stated, shall conform to the general requirements of EN 932-5.

5.2.2 Sampling apparatus**.**

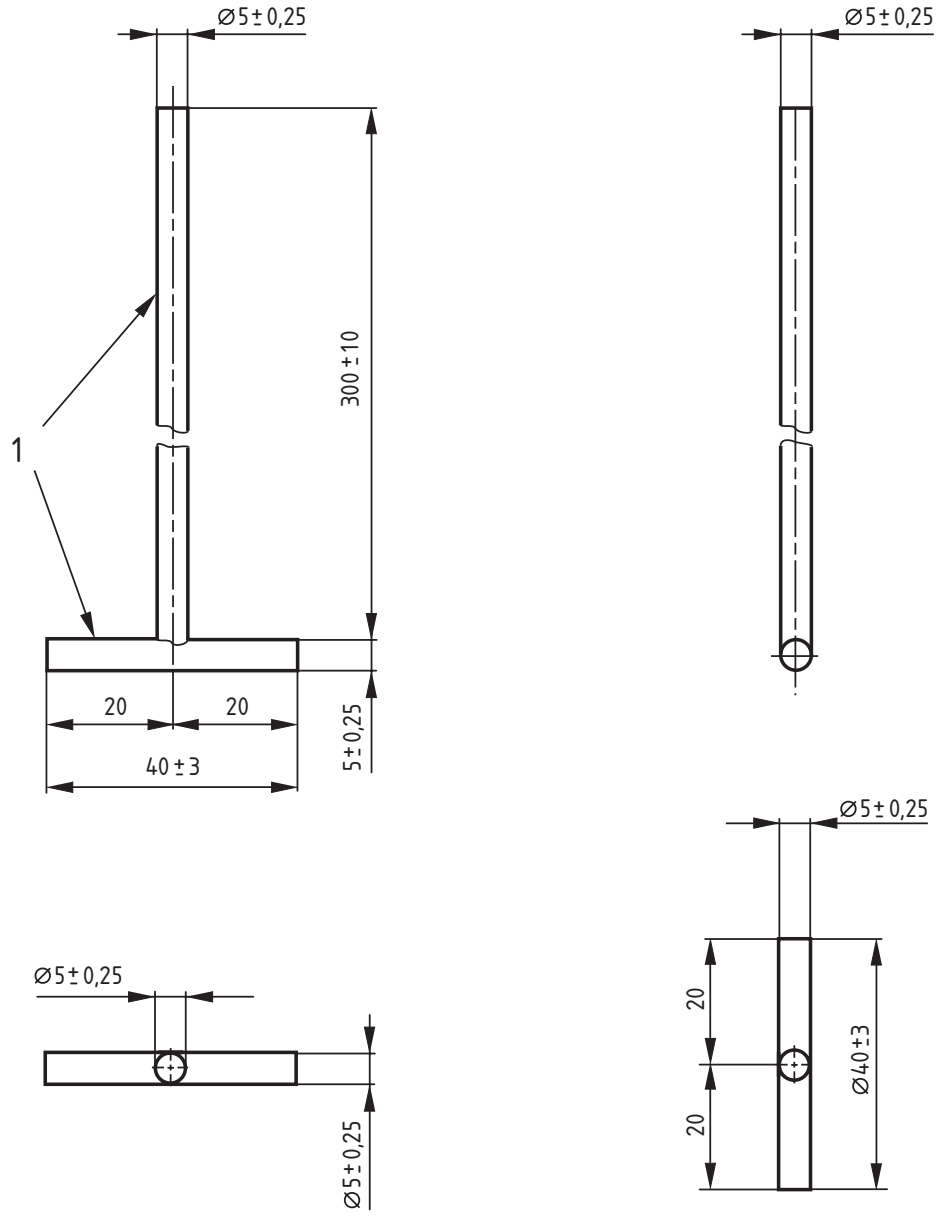
5.2.3 A balance capable of weighing up to 2000 g, accurate to 0,1 g. Other analytical balance capable of weighing with an accuracy of 1 mg.

5.2.4 *Glass conical flask*, wide-mouthed, 250 ml capacity.

5.2.5 *Water bath*, capable of maintaining a temperature of (60 ± 1) °C.

5.2.6 *Motor-driven T-shaped stirrer*, capable of maintaining (25 ± 1) revs/s (see Figure 1).

All dimensions in millimetres



Key

1 Welded steel rods

Figure 1 — T-shaped stirrer

5.2.7 *Glass beaker,* 600 ml capacity.

5.2.8 *Spatula.*

5.2.9 *Desiccator.*

5.2.10 *Sieve 0,125 mm*, complying with EN 933-2.

5.2.11 *Well-ventilated oven*, capable of maintaining a temperature of (110 ± 5) °C.

5.2.12 *Graduated measuring cylinder,* 100 ml capacity.

5.2.13 *Two thermometers,* 0 °C to 100 °C with 1 °C sub-divisions.

5.2.14 *Stop-watch*, or timer, readable to 1 s.

5.2.15 Viscometer

NOTE A suitable viscometer is described in EN 12595.

5.2.16 *Büchner funnel,* 90 mm diameter.

5.2.17 *Vacuum flask*, with suitable Büchner funnel adapter.

5.2.18 *Medium grade filter paper*, ashless, for quantitative analysis, of a diameter appropriate to the size of the funnel (5.2.16).

5.3 Sampling

The laboratory sub-sample shall be taken in accordance with EN 932-1 and reduced in accordance with EN 932-2, to produce a mass of about 50 g.

5.4 Preparation of test portions

Dry the reduced mass of filler for 4 h in an oven at a temperature of (110 ± 5) °C and cool to room temperature in a desiccator for at least 90 min. If agglomerates are present in the material, reduce these agglomerates to powder by means of a spatula. Mix the pulverised agglomerates with the rest of the sub-sample and sieve the sub-sample through the 0,125 mm sieve. Remix the material passing the sieve and take (10 ± 0,1) g as the test portion (m_0).

5.5 Procedure

Place (50,0 ± 0,5) g of low viscosity bitumen solution (5.1.3) into the conical flask and add the test portion of filler to the conical flask. Measure (100 ± 5) ml of demineralized water into the measuring cylinder.

Place the conical flask and the measuring cylinder in the water bath until a temperature of (60 ± 1) °C has been reached by the contents of both vessels (checking by means of thermometers) and maintain this temperature during the test. Stir the contents of the conical flask mechanically for (300 ± 5) s and then allow the mixture to stand for (300 ± 5) s.

Pour the water from the measuring cylinder into the conical flask and stir again for (300 ± 5) s.

Examine the mixture to see if uncoated filler has separated. If not, the filler shall be considered as non susceptible to water.

If the filler separates, or in the case of turbidity of the water, determine the water susceptibility as follows.

Allow the conical flask and its contents to cool so that the mixture becomes kneadable. Pour the water and the filler separated from the mixture by kneading in the flask with a spatula and washing with water. Pour the water and the separated filler into the beaker and repeat the process until the washing with water becomes clear.

Weigh a dried filter paper and records its mass (m_1). Filter the contents of the beaker through the weighed filter paper in a Büchner funnel to the vacuum flask. Remove the last traces of bitumen by washing with kerosene (5.1.2.). Dry the filter and recovered filler in an oven at (110 ± 5) °C to constant mass.

Weigh the filter paper with the filler to the nearest 1 mg (m_2).

5.6 Calculation and expression of results

Calculate the water susceptibility (W_s), as a percentage by mass of the filler, in accordance with the following equation

$$W_s = \frac{m_2 - m_1}{m_0} \times 100 \quad (1)$$

Where :

m_o is the mass of the test portion in grams;

m_1 is the mass of the filter paper in grams;

m_2 is the mass of the filter paper and filler in grams.

W_s should be rounded to the nearest 1 % by mass.

5.7 Test report

The test result shall be accompanied by an affirmation that the water susceptibility was determined in accordance with this European Standard.

The test report shall include the following additional information:

a) source, type and sizes of aggregate submitted for test ;

b) results of water susceptibility.

Annex A
(normative)

Determination of the volume increase and loss of stability of a Marshall specimen

A.1 General

This annex specifies a method for the determination of the volume increase and loss of stability of a Marshall specimen (VIM) which gives a measure of the influence of fillers on the durability of an asphalt in the presence of water.

A.2 Principle

Hot mix asphalt 0/8, consisting of aggregates suitable for use and incorporating the filler under test, shall be tested to determine volume increase and loss of stability after storage in water (40 ± 1) °C for 48 h. The increase in volume shall be indicated in %.

A.3 Materials

A.3.1 *Bitumen* **:** 160/220

A.3.2 Aggregates:

Coarse aggregate shall be volumetrically stable under the conditions of this test.

Aggregate sizes:

a) 5/8 (Flakiness Index FI_{20}or Shape Index SI_{20}, determined in accordance with EN 933-3 or EN 933-4 respectively)

b) 2/5

c) 0,125/2

A.3.3 *Test filler***,** < 0,125 mm

A.4 Apparatus

A.4.1 *All apparatus,* unless otherwise stated, shall conform to the general requirements of EN 932-5.

A.4.2 *Apparatus for preparation of a Marshall specimen*, as specified in EN 12697-30.

A.4.3 Water bath to enable the samples to be held at (40 ± 1) °C and (25 ± 1) °C. The water bath shall have a grid which enables the samples to be surrounded by water on all sides. It shall be large enough to ensure that the amount of water available is equal to at least three times the volume of the samples.

A.4.4 Balance capable of weighing up to 5 000 g, accurate to 0,5 g, with a device for weighing under water (e.g. wire basket).

A.4.5 Desiccator

A.4.6 Vacuum or water-jet pump.

A.4.7 Mixer capable of producing mix sufficient for 8 Marshall-specimens in accordance with EN 12697-35.

A.5 Sampling

Samples shall be taken in accordance with EN 932-1 and reduced in accordance with EN 932-2.

The masses of the test portions for Marshall specimens are specified in Table A.1.

Table A.1 — Mass of test portions

Aggregate sizes (d/D) mm	Mass of test portion kg
5/8	3
2/5	3
0,125/2	4,5
0/0,125 (test filler)	1,5

Dry the reduced mass of filler for 4 h in an oven at a temperature of (110 ± 5) °C and cool to room temperature in a desiccator for at least 90 min. Sieve it on 125 µm.

A.6 Preparation of Marshall specimens

Screen the coarse aggregate to remove oversize and undersize and wash to remove fines. Screen the fine aggregate into size fractions of 0,125/0,25 mm, 0,25/0,71 mm and 0,71/2,0 mm by wet sieving.

Eight Marshall specimens shall be prepared in accordance with EN 12697-30 from a mixture of hot mix asphalt using the proportion of aggregates shown in Table A.2.

The bitumen is to be used in a quantity which, together with the aggregate mix specified, gives a void content of (5,5 ± 0,5) % by volume of a Marshall specimen.

Heat the aggregate mixture and bitumen for eight Marshall specimens in an air circulating oven without fresh air supply for about 3 h at 140 °C. Place the hot aggregates into the mixer preheated to 140 °C and add the prescribed quantity of binder. Mix the components in accordance with EN 12697-35 to prepare a mixture to produce eight Marshall specimens.

Directly after the mixing process, the aggregate mixture is divided in 8 subsamples.

Heat the asphalt mixtures to 140 °C, again in the ventilated oven, within 1 h of mixing process.

Subsequently compact the specimens in accordance with EN 12697-34 within 30 min.

Table A.2 — Proportion of the aggregates

Aggregate	Fractions mm	% by mass
Coarse	5/8 2/5	25 25
Fine	0,71/2 0,25/0,71 0,125/0,25	25 11 4
Test filler	0/0,125	10

A.7 Procedure

Determine the volume before storage (V_A) of four specimens in accordance with EN 12697-12.

Directly after water storage determine the volume (V_Q), in accordance with EN 12697-6.

Determine the Marshall stability values in accordance with EN 12697-34.

Determine the Marshall stability values of the four remaining specimens in accordance with EN 12697-34.

A.8 Calculation and expression of results

Calculate the water susceptibility as the percentage increase in volume of Marshall specimens with the test filler after water storage in accordance with the following equation;

$$Q = \frac{V_Q - V_A}{V_A} \times 100 \qquad \text{(A.1)}$$

Where:

Q is the volume increase in %;

V_A is the volume of specimens before water storage, in cubic centimetres;

V_Q is the volume of specimen after water storage, in cubic centimetres.

The volume increase shall be reported to the nearest 0,1 % by volume as the mean of a minimum of three individual values.

The highest individual value minus the lowest individual value should not exceed 25 % of the mean of the individual values (or 25 % by volume if this is greater).

To determine the mean value, only those values which fall within the permitted range may be used. Furthermore the mass loss between the dry mass before test and the dry mass after test shall not exceed 2 g.

Calculate the loss of stability (S_{MA}) in accordance with the following equation:

$$S_{MA} = \frac{S_M - S_{MQ}}{S_M} \times 100 \quad \text{(A.2)}$$

Where:

S_{MA} is the stability loss in percentage;

S_M is the stability without the volume increase test in kilo Newton;

S_{MQ} is the stability after the volume increase test in kilo Newton.

Calculate the mean loss of stability and report to the nearest 1 %.

A.9 Test report

The test report shall contain:

a) source, type and sizes of all aggregates submitted for test;

b) results of volume increase and stability loss.

A.10 Precision

Repeatability (r) and reproducibility (R) have been determined by a German round-robin test with 12 laboratories in 1979.

Table A.3 — Repeatability (same observer, same apparatus)

Volume increase			
	Up to 1 % volume	Over 1 % volume	
		% of numerical value of result	% of mean value of result
Standard deviation σ_r	0,09	9	-
Repeatability σ_r x 2,77	0,25	-	25

Table A.4 — Reproducibility (different observers, different apparatus)

Volume increase				
		Up to 1 % volume	Over 1% volume	
			% of numerical value of result	% of mean value of result
Standard deviation σ_r		0,18	18	-
Repeatability σ_r x 2,77		0,50	-	50
Confidence interval ± q_R	For single test result ± q_{R1} = ± 1,96 σ_r	± 0,35	35	-
	For test result as mean of 2 results q_{R2} = ± 1,36 σ_r	± 0,25	-	25
	For test result as mean of 3 results q_{R3} = ± 1,16 σ_r	± 0,20	-	20

Bibliography

NBN B 11-225 : *February 1995, Fillers pour mélanges hydrocarbonés — Détermination de la sensibilité à l'eau (projet)*

NEN 3982 : March 1992, *Vulstoffen voor bitumineuzemengsels — Bepaling van de watergevoeligheid (2e ontwerp)*

ISO 1000 : 1992, *SI Units and recommendations for the use of their multiples and of certain other units*

EN 196-6, *Methods of testing cement - Determination of fineness*

EN 12595, *Bitumen and bituminous binders — Determination of kinematic viscosity*

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: +44 (0)20 8996 9000. Fax: +44 (0)20 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: +44 (0)20 8996 9001. Fax: +44 (0)20 8996 7001. Email: orders@bsi-global.com. Standards are also available from the BSI website at http://www.bsi-global.com.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: +44 (0)20 8996 7111. Fax: +44 (0)20 8996 7048. Email: info@bsi-global.com.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: +44 (0)20 8996 7002. Fax: +44 (0)20 8996 7001. Email: membership@bsi-global.com.

Information regarding online access to British Standards via British Standards Online can be found at http://www.bsi-global.com/bsonline.

Further information about BSI is available on the BSI website at http://www.bsi-global.com.

Copyright

BSI
389 Chiswick High Road
London
W4 4AL