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Tests for chemical properties of aggregates

Part 4: Determination of water susceptibility of fillers for bituminous mixtures

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

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

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

This European Standard was approved by CEN on 22 November 2021.

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European foreword

This document (EN 1744-4:2021) 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of an identical text or by endorsement, at the latest by June 2022, and conflicting national standards shall be withdrawn at the latest by June 2022.

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

This document supersedes EN 1744-4:2005.

This document 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*

— *Part 7: Determination of loss on ignition of Municipal Incinerator Bottom Ash Aggregate (MIBA Aggregate)*

— *Part 8: Sorting test to determine metal content of Municipal Incinerator Bottom Ash (MIBA) Aggregates*

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This document 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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 933-1:2012, *Tests for geometrical properties of aggregates - Part 1: Determination of particle size distribution - Sieving method*

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 12274-7:2005, *Slurry surfacing - Test Methods - Part 7: Shaking abrasion test*

EN 12591, *Bitumen and bituminous binders – Specifications for paving grade bitumen*

EN 12697-6:2020, *Bituminous mixtures - Test methods - Part 6: Determination of bulk density of bituminous specimens*

EN 12697-12:2018, *Bituminous mixtures - Test methods - Part 12: Determination of the water sensitivity of bituminous specimens*

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

EN 12697-34, *Bituminous mixtures - Test methods - Part 34: Marshall test*

EN 12697-35, *Bituminous mixtures - Test methods - Part 35: Laboratory mixing*

EN 12846-2, *Bitumen and bituminous binders - Determination of efflux time by the efflux viscometer - Part 2: Cut-back and fluxed bituminous binders*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth (ISO 3310-1)*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

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 1 to entry: 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 1 to entry: 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 1 to entry: 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.

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 ((60 ± 5) s, S.T.V. (Standard Tar Viscometer) 10 mm) as specified in EN 12846-2.

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.

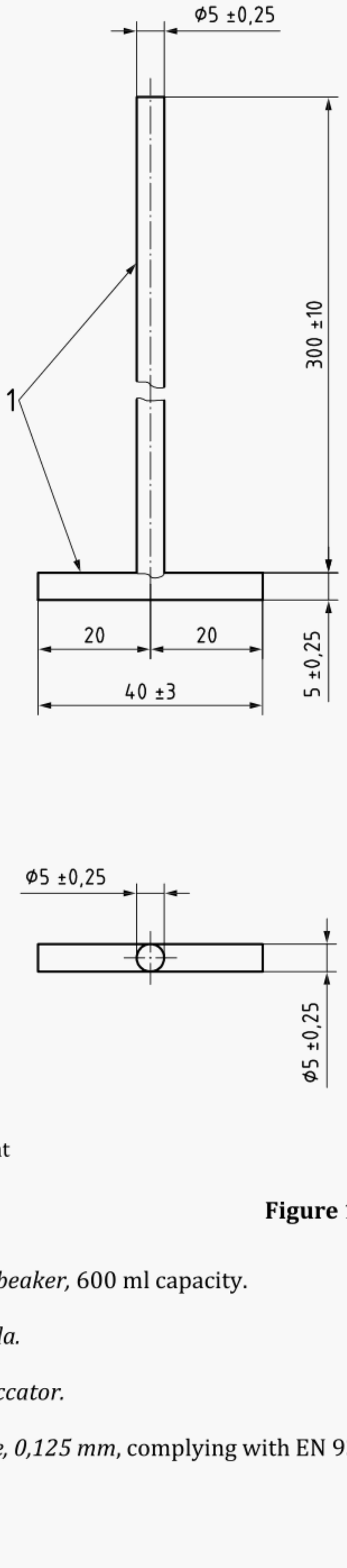
5.2.4 *Glass conical flask*, wide-mouthed, or disposable tinplate can. Glass or tinplate can, shall be of 250 ml capacity.

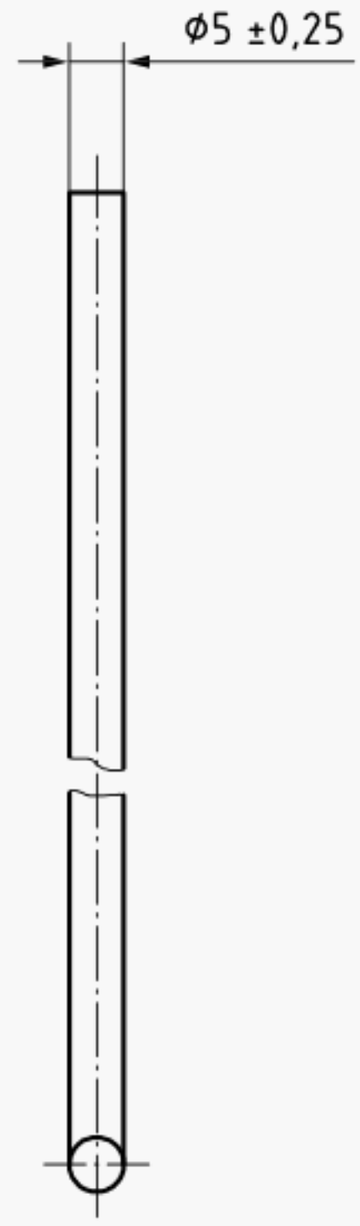
5.2.5 *Water bath*, capable of maintaining a temperature of (60 ± 1) °C.

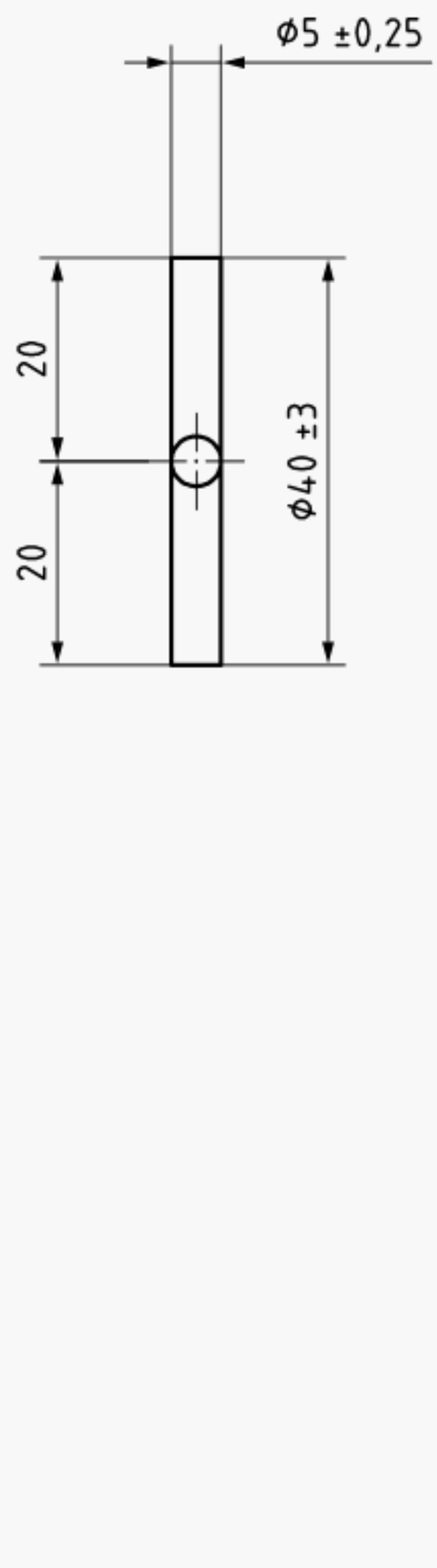
5.2.6 *Motor-driven T-shaped stirrer*, capable of maintaining (25 ± 1) revs/s (see Figure 1).

5.2.7 *Lead cap with a hole in its centre to cover the tinplate can as the can is light-weighted with risk to float and to allow stirring.*

Dimensions in millimetres







Key

1 Air vent

Figure 1 — T-shaped stirrer

5.2.8 *Glass beaker,* 600 ml capacity.

5.2.9 *Spatula.*

5.2.10 *Desiccator.*

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

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

5.2.13 *Graduated measuring cylinder,* 100 ml capacity.

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

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

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 in an oven at a temperature of (110 ± 5) °C to constant mass 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 (m0).

5.5 Procedure

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

Place the conical flask or the tinplate with the lead cap 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 or the tinplate can with its lead cap 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 or the tinplate can with the lead cap and stir again for (300 ± 5) s.

In the case of use of tinplate can, the supernatant water is to be transferred into a beaker, to see if uncoated filler has separated.

In the case of a use of a glass conical flask, 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 or the tinplate can 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_0 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 document.

The test report shall include the following information:

a) reference to this document;

b) identification of the test sample, including identification of the source and date of sampling;

c) sample reception date if different from sampling date;

d) identification of the laboratory;

e) the test results for water susceptibility;

f) deviations from the reference method – if any.

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*:

A.3.2.1 Aggregate stability:

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

A.3.2.2 Aggregate size/shape:

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

b) 2/5 mm;

c) 0,125/2 mm.

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 V*acuum 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	25
	2/5	25
Fine	0,71/2	25
	0,25/0,71	11
	0,125/0,25	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 shortage, 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 the following information:

a) reference to this document;

b) identification of the test sample, including identification of the source and date of sampling;

c) sample reception date if different from sampling date;

d) identification of the laboratory;

e) results of volume increase and stability loss;

f) deviations from the reference method – if any.

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 $\sigma_r \times 2,77$	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	9	—
Repeatability $\sigma_r \times 2,77$		0,50	—	50
Confidence interval $\pm q_R$	For single test result $\pm q_{R1} = \pm 1,96\sigma_r$	±0,35	35	—
	For test result as mean of 2 results $q_{R2} = \pm 1,36\sigma_r$	±0,25	—	25
	For test result as mean of 3 results $q_{R3} = \pm 1,16\sigma_r$	±0,20	—	20

Annex B
(informative)

Water susceptibility of fillers – Shaking abrasion method

B.1 General

This annex specifies the method for the determination of the water susceptibility of fillers for bituminous mixtures using the shaking abrasion method. The method is based on the procedure pursuant to EN 12274-7 “Slurry surfacing – Test Methods – Part 7: Shaking abrasion test”.

B.2 Principle

The shaking abrasion test determines the water susceptibility of an asphalt mixture – prepared using fillers, fine aggregates and bitumen. The test measures the abrasion of standardized specimens that occurs when cylindrical specimens of a compacted asphalt mixture are placed in water-filled shaking cylinders, which are rotated around an axis in a suitable device with overhead movement. In addition, the cylindrical specimens are tested for swelling as a result of being stored in water.

Three cylindrical specimens measuring 30 mm in diameter are tested in each series of tests. They are prepared from a standardized specimen mixture using a standard bitumen. The specimens are statically compacted.

B.3 Apparatus

B.3.1 Conical steel bowl, 18 cm in diameter.

B.3.2 Porcelain crucible 1000 ml with handle.

B.3.3 3x porcelain crucible 125 ml with handle.

B.3.4 Porcelain pestle, 57 mm in diameter, 180 mm in length.

B.3.5 Stirring thermometer up to 220 °C.

B.3.6 Heater with air bath.

B.3.7 Heating chamber with air circulation, temperature controlled, for maintaining a temperature of (110 ± 5) °C and (150 ± 5) °C.

B.3.8 Test sieves with metal mesh pursuant to ISO 3310-1 with opening widths of 0,4 mm, 0,25 mm and 0,125 mm.

B.3.9 Compaction moulds with base plates pursuant to EN 12274-7 (at least 3 per series of tests).

B.3.10 Compaction pistons, one per compaction mould pursuant to EN 12274-7.

B.3.11 Filling funnel pursuant to EN 12274-7.

B.3.12 Thermostatic water bath (EN 12697-12:2018, Clause 5, Method A).

B.3.13 Vacuum system (EN 12697-12:2018, Clause 5, Method A).

B.3.14 Compression test machine with a suitable load range and a feed rate of (20 ± 3) mm/min.

B.3.15 Shaking device equipped with at least 3 shaking cylinders (see Figure B.1 and EN 12274-7:2005, Figure 4).

B.3.16 Shaking cylinder with sealing cap (see EN 12274-7:2005, Figures 4 and 5).

B.3.17 Chamois cloth.

B.3.18 Hardboard, approx. 1 cm thick.

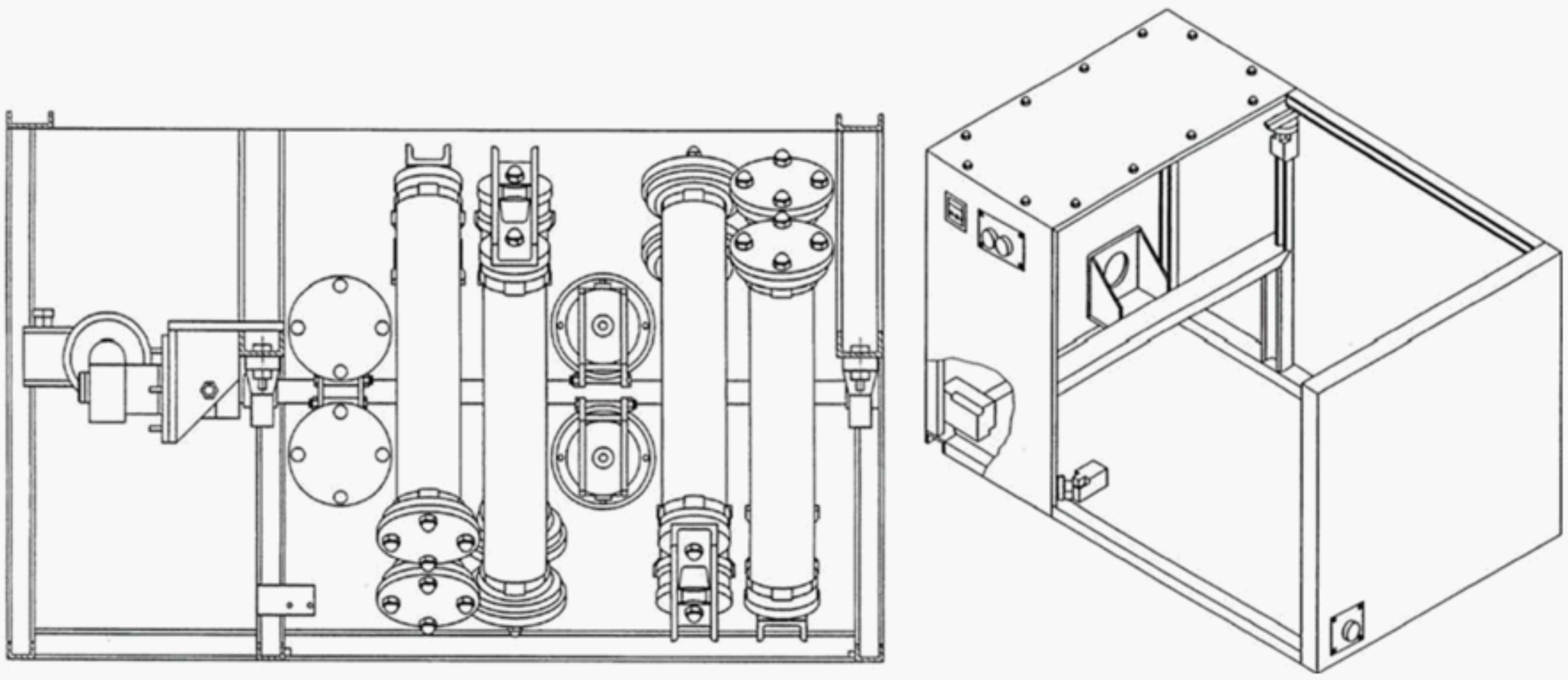


Figure B.1 — Shaking device

B.4 Materials

B.4.1 Binder.

The binder used for these tests is a bitumen 70/100 to EN 12591 “Bitumen and bituminous binders – Specifications for paving grade bitumen”. The reference for the applied bitumen can be found at www.fgsv.de/bezugsadressen.

B.4.2 Aggregate.

The fine aggregate 0/0,4 mm is composed of standard sands (“FH31” and “F36”). The reference addresses for the applied standard sands can be found at www.fgsv.de/bezugsadressen.

The aggregates shall be dried at (110 ± 5) °C inside the heating chamber.

Drying aggregates with an open flame or an IR radiator is not permitted.

The unwashed aggregates are divided by dry sieving pursuant to EN 933-1:2012, 7.2 into the particle size classifications 0/0,125; 0,125/0,25; 0,25/0,4 mm.

B.5 Preparation of the specimens

B.5.1 Composition of the specimen mixture

The specimen mixture shall consist of 0/0,4 mm aggregate and standard bitumen 70/100 to EN 12591 and standard sands (“FH31” and “F36”) are used. The reference addresses for the applied bitumen and the applied standard sands can be found at www.fgsv.de/bezugsadressen.

For the test, the aggregate shall have the following composition:

Table B.1 — Proportion of particle size classifications applied in the specimen mixture when determining water susceptibility using the shaking abrasion method

Particle size classification [mm]	**Proportion** [% by mass]
0,25 – 0,4	25
0,125 – 0,25	50
0 – 0,125	25

The particle size classification 0/0,125 mm consists exclusively of the filler to be tested, the particle size range > 0,125 mm consists exclusively of the standard sand.

Table B.2 — Specimen mixture composition for determining water susceptibility using the shaking abrasion method

Particle size classification [mm]	**Net weight of aggregate** [g]
0,25 – 0,4	60 ± 0,2
0,125 – 0,25	120 ± 0,2
0 – 0,125	60 ± 0,2

The quantity of binder B (±0,1 g) is determined as a function of the bulk density of the aggregate 0/0,4 as follows:

$$B = \frac{12,0 \times 2,700}{\rho_g} \left[g \right]$$

where:

B is the quantity of binder, rounded to the nearest 0,1 g;

ρ_g is the bulk density of the aggregate with a composition according to Table B.2 in g/cm³ and calculated according to B.6.3.

The required mass of aggregate and standard bitumen is composed for the required three specimens.

Additional test quantities need to be obtained according to Table B.2 to determine the densities.

B.5.2 Preparation of the specimen mixture

When heating the aggregate to the mixing temperature, the homogenized, weighed aggregate is placed in a steel bowl for (180 ± 10) minutes inside the heating chamber at a temperature of (150 ± 5) °C.

The respective portion of cold binder is weighed into a porcelain crucible and then covered and placed inside the same heating chamber for (18 ± 2) minutes before the end of the aggregate tempering period.

Alternatively, the binder can be tempered in a hot air bath. The binder shall not be heated more than once. Aluminium foil or a metal lid can be used to cover the binder.

The heated aggregates are then added to the binder in the porcelain crucible, pre-mixing at the same time with the stirring thermometer. Pre-mixing, including pouring, shall be carried out for (30 ± 3) seconds.

Immediately afterwards, the porcelain crucible is placed on the hardboard to reduce heat loss and the asphalt mixture is kneaded (about one stirring rotation per second) with the heated porcelain pestle for (150 ± 10) seconds without the application of heat. The mixture, which has cooled slightly as a result of kneading, is then heated to (150 ± 5) °C over the heater with air bath, while stirring continuously with the stirring thermometer. Subsequently, the mixture is then kneaded for a second time on the hardboard with the porcelain pestle for (150 ± 10) seconds without the application of heat.

Once the specimen mixture has cooled down to below 50 °C, the quantities of mixture required for each specimen are weighed into individual porcelain crucibles (125 ml). The net weight is (40 ± 0,3) g.

B.5.3 Manufacturing the specimens

The specimens are prepared on the day of mixture preparation. The asphalt mixture specimens, which have cooled down to room temperature, are placed in the small porcelain crucibles inside the heating chamber tempered to (150 ± 5) °C. The compaction moulds and pistons with their base plates are also heated here. After a tempering period of (30 ± 2) minutes, only the first specimen is removed from the heating chamber. The remaining specimens and their moulds remain inside the heating chamber. The removed specimen is poured through the filling funnel into the heated compaction mould. Pre-compaction is achieved by lightly tapping the filled compaction mould on the hardboard. The compaction piston is then placed on the pre-compacted specimen, and the compaction mould with piston is placed in the compression test machine. Compaction takes place at a feed rate of (20 ± 3) mm/min until a force of (10 ± 0,5) kN is reached. After the aforesaid force has been reached, the specimen shall be immediately relieved of the force.

The second and third specimens are prepared in the same way as the first specimen. This process shall be carried out quickly to ensure compaction of the third specimen is completed no more than 10 min after the start of compaction of the first specimen.

After being air cooled to a temperature of 40 to 80 °C, the specimens are pushed downwards and demoulded, i.e. pushed out of the other end of the slightly conical mould cylinders.

NOTE 1 Demoulding can be done using, e.g. a light hand press or a vice.

The three specimens are stored in air for (24 ± 3) hours at (25 ± 2) °C after demoulding.

Before any further tests are carried out, protruding burrs shall be removed from the upper and lower edges of the specimens. The sand asphalt specimens obtained in this way are designated P1 to P3 for each series.

NOTE 2 Fine sandpaper is suitable for deburring.

B.6 Procedure

B.6.1 Determining water absorption and swelling

The dry mass (m_p) of the three specimens (P1, P2 and P3) shall be determined to the nearest 0,01 g at the start of the test.

The specimens (P1, P2 and P3) are to be stored at (25 ± 2) °C in a water bath. After being stored in water for (90 ± 30) minutes, they are weighed under water (m_{WA}) and in air (m_{LA}) as specified in EN 12697-6:2020, Method B, Steps d), e) and g) to the nearest 0,01 g. The difference between the determined masses (m_{LA} - m_{WA}) is equal to the numerical value of the initial volume of the specimen V_A in cm^3.

Afterwards, the specimens P1, P2 and P3 are treated pursuant to EN 12697-12:2018, 6.1.2.2.1 to 6.1.2.2.4.

For this purpose, the specimens are stored on a tray with a perforated intermediate base in distilled water tempered to (20 ± 5) °C inside a vacuum chamber. The specimens shall be covered at least 20 mm with distilled water.

The vacuum chamber shall be evacuated to a residual pressure of (6,7 ± 0,3) kPa within (10 ± 1) minutes. This pressure is to be maintained for a period of (30 ± 5) minutes. After which, the atmospheric pressure should be allowed to build up again in the vacuum chamber.

The tray with the specimens is then removed from the vacuum system, transferred to the water bath and stored there for (90 ± 30) minutes at (25 ± 2) °C. After storage, the specimens are to be weighed under water (m_{WV}) and in air (m_{LV}) according to the aforesaid procedure. The difference between the determined masses (m_{LV} - m_{WV}) is equal to the numerical value of the volume after water absorption of the specimen V_V in cm^3.

Subsequently, the specimens P1 to P3 are stored for (144 ± 6) hours in a water bath at (25 ± 2) °C.

After being stored in water, the specimens (P1, P2 and P3) are to be weighed again under water (m_{WQ}) and in air (m_{LQ}) as specified in EN 12697-6:2020, Method B, Steps d), e) and g) to the nearest 0,01 g. The difference between the two weighing results is used to determine the volume after swelling V_Q, similar to the V_A calculation procedure.

B.6.2 Determining the shaking abrasion

The test shall be carried out immediately after determining the volume after swelling V_Q (5.1) on specimens P1, P2 and P3. Each specimen is to be placed in one of the shaking cylinders previously filled with (750 ± 5) ml of fresh drinking water at a temperature of (25 ± 2) °C. Afterwards, the shaking cylinders are fitted with the sealing caps and inserted into the shaking device. The shaking device is then operated at a speed of (20 ± 2) rpm at room temperature until completing a total of (3600 ± 10) rotations. The specimens are to be extracted from the cylinders and any loose components immediately removed under running water. After dabbing the specimens with the damp chamois leather, the mass is determined to the nearest 0,01 g after exposure to shaking abrasion (m_s).

There shall be no residual solvent in the shaking cylinders during the test.

B.6.3 Calculation and expression of results

B.6.3.1 Bulk density of the composite aggregate 0/0,4 mm

The bulk density of the composite aggregate 0/0,4 mm is calculated using the following equation:

$$\rho_g = \frac{100}{\frac{P_{a1}}{\rho_{a1}} + \frac{P_{a2}}{\rho_{a2}}} \left[g / cm^3 \right]$$

where:

ρ_g is the bulk density of the composite aggregate 0/0,4 mm of a series, reported to the nearest 0,001 g/cm^3;

P_{a1} is the percentage of the aggregate 0,125/0,4 in the aggregate 0/0,4 mm (in % by mass) according to Table B.1;

ρ_{a1} is the bulk density of the aggregate 0,125/0,4, reported to the nearest 0,001 g/cm^3;

P_{a2} is the percentage of the filler 0/0,125 in the aggregate 0/0,4 mm (in % by mass) according to Table B.1;

ρ_{a2} is the bulk density of the filler 0/0,125, reported to the nearest 0,001 g/cm^3.

B.6.3.2 Water absorption

The water absorption (W_V) is calculated in line with EN 12274-7:2005, Clause 6.1.2 and is to be stated, rounded to the nearest 0,1 % by volume, as the arithmetic mean of three individual values (P1, P2 and P3):

$$W_V = \frac{m_{LV} - m_p}{m_{LA} - m_{WA}} \times 100 \left[\%\ \text{by volume}\right] \text{ if } V_V \leq V_A$$

$$W_V = \frac{(m_{WV} - m_p) + (m_{LA} - m_{WA})}{m_{LA} - m_{WA}} \times 100 \left[\%\ \text{by volume}\right] \text{ if } V_V > V_A$$

where:

W_V is the water absorption, in % by volume;

m_{LA} is the mass of the specimen in air before the application of vacuum, in g;

m_{WA} is the mass of the specimen under water before the application of vacuum, in g;

m_{LV} is the mass of the specimen in air after the application of vacuum, in g;

m_{WV} is the mass of the specimen under water after the application of vacuum, in g;

m_p is the mass of the specimen in air before the test, in g;

V_A corresponds approximately to $m_{LA} - m_{WA}$, in g – the volume of the specimen before water absorption, in cm^3;

V_V corresponds approximately to $m_{LV} - m_{WV}$, in g – the volume of the specimen after water absorption, in cm^3.

B.6.3.3 Swelling

The swelling (Q), reported to nearest 0,1 % by volume, is to be stated as the arithmetic mean of the three individual values (P1, P2 and P3):

$$Q = \frac{V_Q + V_A}{V_A} \times 100 \left[\%\ \text{by volume}\right]$$

where:

Q is the swelling, in % by volume;

V_Q is the volume of the specimen after swelling, in cm^3;

V_A is the initial volume of the specimen, in cm^3.

B.6.3.4 Shaking abrasion

The shaking abrasion (S_A), reported to the nearest 0,1 % by mass, is to be stated as the arithmetic mean of the three individual values (P1, P2 and P3):

$$S_A = \frac{m_{LQ} - m_s}{m_{LQ}} \times 100 \left[\% \text{ by mass}\right]$$

where:

S_A is the shaking abrasion of the specimen, rounded to the nearest 0,1 % by mass;

m_{LQ} is the mass of the specimen in air after storage in water, rounded to the nearest 0,01 g;

m_s is the mass of the specimen after exposure to shaking-abrasion, rounded to the nearest 0,01 g.

B.7 Test report

The test report shall contain the following information:

a) reference to this document;

b) identification of the test sample, including identification of the source and date of sampling;

c) Designation of the particle size fraction/particle size group from which the test particle was obtained;

d) sample reception date if different from sampling date;

e) identification of the laboratory;

f) results of:

   i. Water absorption (W);

   ii. Swelling (Q);

   iii. Shaking abrasion (S_A).

g) deviations from the reference method – if any.

The results are easily expressed in tabular form.

Table B.3 — Example of tabular representation

Test feature	Result	
Water absorption (W)		% by volume
Swelling (Q)		% by volume
Shaking abrasion (S_A)		% by mass

Bibliography

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

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