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

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

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

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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 COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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EN 1744-4:2021 (E)

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of an identical text or by endorsement, at the latest by June 2022, and conflicting national standards shall be withdrawn at the latest by June 2022.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* [SIST EN 1744-4:2022](https://standards.iteh.ai/catalog/standards/sist/64b7dbd1-1091-4186-b037-715428612141/en-1744-4-2022)
- *Part 2: Determination of resistance to alkali/aggregate reaction* <https://standards.iteh.ai/catalog/standards/sist/64b7dbd1-1091-4186-b037-715428612141/en-1744-4-2022>
- *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)*

EN 1744-4:2021 (E)

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*.

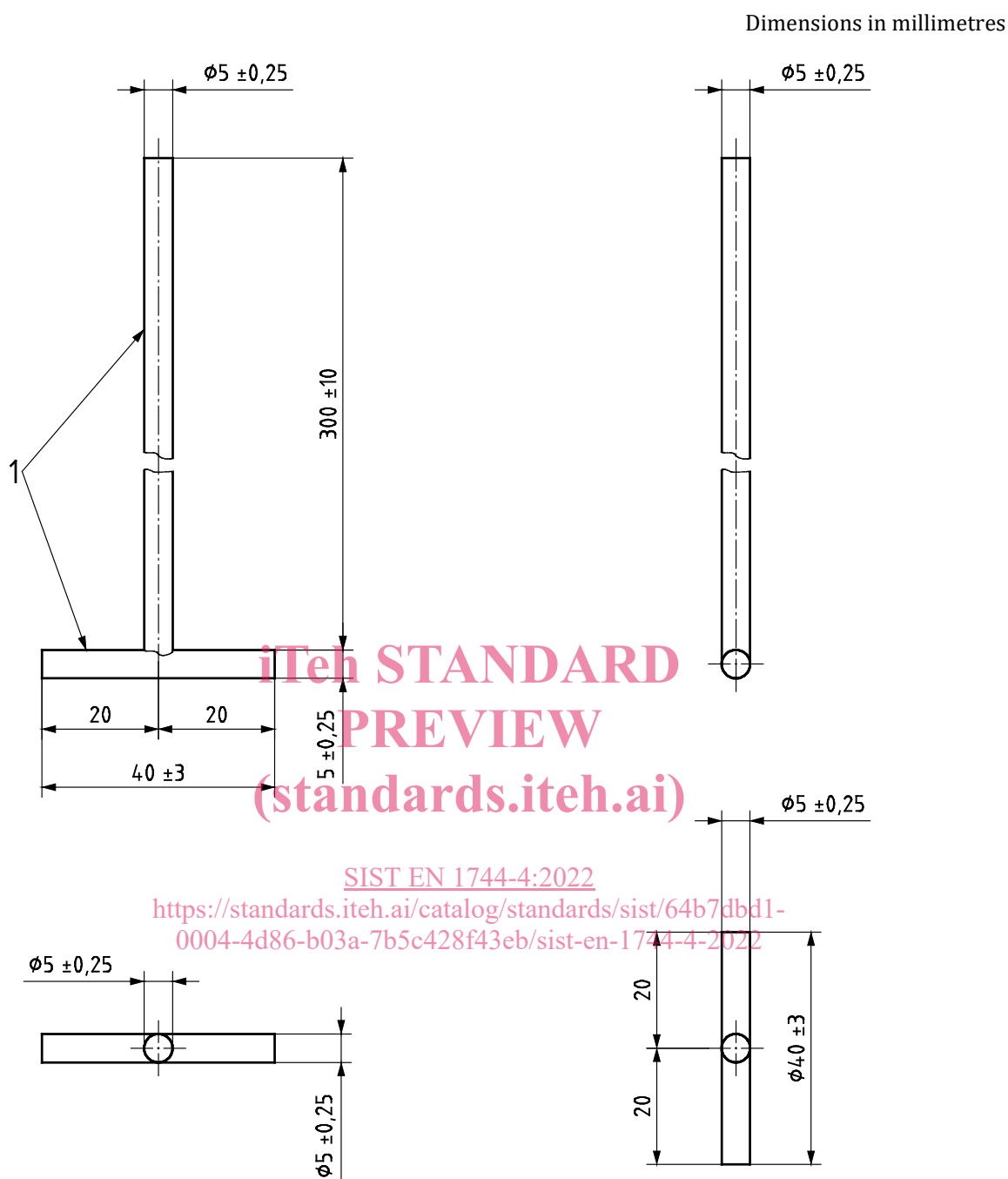
5.2.3 A *balance* capable of weighing up to 2000 g, accurate to 0,1 g. Other analytical balance capable of weighing with an accuracy of 1 mg.

5.2.4 *Glass conical flask*, wide-mouthed, or disposable tinfoil can. Glass or tinfoil 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 tinfoil can as the can is light-weighted with risk to float and to allow stirring*.

**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 $(10 \pm 0,1)$ g as the test portion (m_0).

5.5 Procedure

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

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

In the case of use of tinfoil 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 tinfoil 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