



BSI Standards Publication

Unbound and hydraulically bound mixtures

Part 54: Test method for the determination of frost susceptibility — Resistance to freezing and thawing of hydraulically bound mixtures

National foreword

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

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Ungebundene und hydraulisch gebundene Gemische - Teil 54: Prüfverfahren zur Bestimmung der Frostempfindlichkeit - Frost-Tau-Wechselbeständigkeit von hydraulisch gebundenen Gemischen

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Foreword

This document (CEN/TS 13286-54:2014) has been prepared by Technical Committee CEN/TC 227 "Road materials", the secretariat of which is held by DIN.

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This Technical Specification is one of a series of standards as listed below:

EN 13286-1, *Unbound and hydraulically bound mixtures — Part 1: Test methods for laboratory reference density and water content — Introduction, general requirements and sampling*

EN 13286-2, *Unbound and hydraulically bound mixtures — Part 2: Test methods for the determination of laboratory reference density and water content — Proctor compaction*

EN 13286-3, *Unbound and hydraulically bound mixtures — Part 3: Test methods for laboratory reference density and water content — Vibrocompression with controlled parameters*

EN 13286-4, *Unbound and hydraulically bound mixtures — Part 4: Test methods for laboratory reference density and water content — Vibrating hammer*

EN 13286-5, *Unbound and hydraulically bound mixtures — Part 5: Test methods for laboratory reference density and water content — Vibrating table*

EN 13286-7, *Unbound and hydraulically bound mixtures — Part 7: Cyclic load triaxial test for unbound mixtures*

EN 13286-40, *Unbound and hydraulically bound mixtures — Part 40: Test method for the determination of the direct tensile strength of hydraulically bound mixtures*

EN 13286-41, *Unbound and hydraulically bound mixtures — Part 41: Test method for the determination of the compressive strength of hydraulically bound mixtures*

EN 13286-42, *Unbound and hydraulically bound mixtures — Part 42: Test method for the determination of the indirect tensile strength of hydraulically bound mixtures*

EN 13286-43, *Unbound and hydraulically bound mixtures — Part 43: Test method for the determination of the modulus of elasticity of hydraulically bound mixtures*

EN 13286-44, *Unbound and hydraulically bound mixtures — Part 44: Test method for the determination of the alpha coefficient of vitrified blast furnace slag*

EN 13286-45, *Unbound and hydraulically bound mixtures — Part 45: Test method for the determination of the workability period of hydraulically bound mixtures*

EN 13286-46, *Unbound and hydraulically bound mixtures — Part 46: Test method for the determination of the moisture condition value*

EN 13286-47, *Unbound and hydraulically bound mixtures — Part 47: Test method for the determination of the California bearing ratio, immediate bearing index and linear swelling*

EN 13286-48, *Unbound and hydraulically bound mixtures — Part 48: Test method for the determination of the degree of pulverization*

EN 13286-49, *Unbound and hydraulically bound mixtures — Part 49: Accelerated swelling test of soil treated by lime and/or hydraulic binders*

EN 13286-50, *Unbound and hydraulically bound mixtures — Part 50: Methods for making test specimens using proctor equipment or vibrating table compaction*

EN 13286-51, *Unbound and hydraulically bound mixtures — Part 51: Methods for making test specimens by vibrating hammer compaction*

EN 13286-52, *Unbound and hydraulically bound mixtures — Methods for making test specimens — Part 52: Making specimens by vibro-compression*

EN 13286-53, *Unbound and hydraulically bound mixtures — Methods for making test specimens — Part 53: Making cylindrical specimens by axial compression*

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Introduction

At present, this test method is not proposed as an European Standard (EN) but issued instead as a Technical Specification (TS). It will remain a TS until more data and experience is forthcoming regarding the most appropriate conditioning of specimens immediately prior to freeze-thaw cycling. Reference to the test method reveals that three methods of conditioning are described/permitted for the two day stage between first stage curing (to permit adequate strength development) and the freeze-thaw testing stage. This second stage conditioning can be either complete submersion in a water bath, storage in a humidity cabinet, or continuation of the first stage curing. Typically the latter consists of curing to prevent loss of moisture. As is stated in the test method, it is probable that water bath curing of the test specimens is more robust than humidity cabinet curing which in turn may be more robust than the initial first stage curing. The choice of method is deliberately left for determination at the place of use and will depend on the type and nature of the hydraulically bound mixture or hydraulically treated soil, the particular application and the known/expected climatic conditions. Users of the test method are invited/encouraged to trial the different methods of second stage curing and to provide feedback. Then and only then will consideration be given to specifying a preferred/necessary method of second stage curing and then to the issuing of the TS as a EN.

1 Scope

This Technical Specification specifies a test method for the determination of the resistance of a hydraulically bound mixture to the cyclic action of freezing and thawing.

The method described is suitable for hydraulically bound mixtures, including hydraulically stabilised soils, in accordance with EN 14227-1 to EN 14227-5 and the range of strengths covered by that standard.

When required, a method for determining the change in height of a hydraulically bound subject to freeze thaw is specified in Annex A (normative).

When required, a method for determining the freeze thaw resistance of a hydraulically bound mixture in the presence of salt is specified in Annex B (normative).

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

EN 1367-1, *Tests for thermal and weathering properties of aggregates - Part 1: Determination of resistance to freezing and thawing*

EN 1367-4, *Tests for thermal and weathering properties of aggregates - Part 4: Determination of drying shrinkage*

EN 1367-6, *Tests for thermal and weathering properties of aggregates - Part 6: Determination of resistance to freezing and thawing in the presence of salt (NaCl)*

EN 14227 (all parts), *Hydraulically bound mixtures — Specifications*

3 Terms and definitions

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

3.1 test specimen

sample used in a single determination when a test method requires more than one determination of a property

3.2 laboratory sample

reduced sample derived from a bulk sample for laboratory testing

3.3 hydraulically bound mixture

mixture that sets and hardens by hydraulic reaction

4 Principle

Two sets of three test specimens are prepared from a hydraulically bound mixture. The moulds containing the mixture are then placed in initial (first stage) curing conditions specified by the nature of the hydraulic constituents and in accordance with national regulation or decision at the place of use.

On completion of the first stage curing, the two sets of test specimens (sets A and B) are removed from their moulds and placed in a water bath or humidity cabinet or returned to the initial curing conditions, for two days further curing (second stage).

NOTE 1 Water bath curing is more robust/onerous than humidity cabinet curing which in turn may be more robust/onerous than the initial curing condition employed. The selection therefore of the type of second stage curing depends on the type and nature of the hydraulically bound mixture or hydraulically stabilised soil in question, the particular application for the HBM and the climatic conditions.

On completion of second stage curing, one set of three test specimens (Set A) is removed from the second stage curing, surface dried and tightly wrapped in plastic (cling) film. The second "control" set of three test specimens (Set B) remains in the second stage curing conditions.

Set A specimens are then placed in a prepared freeze thaw cabinet and subjected to ten freeze-thaw cycles, each cycle lasting 24 h. After completion of the tenth freeze thaw cycle, the Set A specimens are unwrapped and returned to the second stage curing regime for one day at 20 °C to ensure complete thawing.

The strength of both sets of three test specimens is then measured and the mean value of strength for each set compared.

NOTE 2 An additional set of three test specimens (Set C) can be used to give additional information about the strength of the mixture after second stage curing. The use of Set C allows the gain in strength of the 'control' set (Set B) between the end of second stage curing and the time of strength testing of Set B to be evaluated.

NOTE 3 When required, the steel hemispherical buttons used to measure the length of the test specimen (Annex A) are also fitted into the inserts at the end of second stage curing.

5 Apparatus

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

5.2 Cylindrical moulds, for test specimen preparation, as specified in the relevant test specimen preparation Standard (see Clause 6). Six moulds are required.

NOTE The use of moulds that are capable of being split along the longitudinal axis may be used to allow removal of the test specimen with the minimum of damage.

5.3 Watertight plastic bags (or plastic film), for initial curing.

5.4 Initial curing area(s), capable of temperature controlled curing at the specified temperature and moisture condition.

A humidity cabinet, capable of control at the specified temperature and 90 % to 100 % relative humidity, is a permitted option.

5.5 Water bath, controlled at (20 ± 2) °C, one of the permitted options for second stage curing.

A humidity cabinet controlled at (20 ± 2) °C and 90 % to 100 % RH is a permitted alternative for second stage curing (as 5.4 above).

5.6 Low temperature cabinet, complying with the requirements of EN 1367-1.

5.7 Electronic thermocouple thermometer, or similar device, accurate to 0,1 °C used to monitor the temperature in the centre of the cooled area in the low temperature cabinet.

The thermocouple probe shall be located in the centre of a metal can complying with EN 1367-1, containing (2 000 ± 5) g of 8/16 mm size aggregate and filled with water to a level at least 10 mm above the aggregate.

5.8 Suitable cloths, to surface dry the saturated test specimens.

5.9 Plastic (cling) film, for tightly wrapping one set of saturated test specimens before they are placed in the low temperature cabinet.

NOTE 1 See Annex A for additional requirements for the base of moulds fitted with inserts for measuring height.

NOTE 2 Suitable apparatus will also be required for test specimen preparation and strength testing, as specified in the relevant European Standards.

NOTE 3 Low temperature cabinets that control the temperature of the thawing stage using air circulation are preferred for this test method.

NOTE 4 Three more moulds are required if a third set of test specimens (Set C) is tested at the end of second stage curing.

6 Test specimens

6.1 Preparation

On day one, make six cylindrical test specimens from a laboratory sample of the hydraulically bound mixture to be tested. Prepare the test specimens in cylindrical moulds using one of the methods from Table 1.

NOTE 1 When required, an additional set of three test specimens (Set C) may be used to give additional information about the strength of the mixture at the end of second stage curing.

NOTE 2 It is important that all test specimens are the same nominal size and are prepared using the same compaction method.

Table 1 — Moulds

| EN 13286 | Method | Diameter mm | Height mm | Mixture size mm |
|----------|-----------------------------|----------------|--------------|--------------------|
| -50 | Proctor and vibrating table | 100 | 120 | Up to 0/22,4 |
| | | 150 | 120 | Up to 0/31,5 |
| -51 | Vibrating hammer | 100 | 100 | Up to 0/22,4 |
| | | 150 | 150 | Up to 0/31,5 |
| -52 | Vibro-compression | 100 | 100 | Up to 0/22,4 |
| | | | 200 | |
| | | 160 | 160 | Up to 0/31,5 |
| | | | 320 | |
| -53 | Axial compression | 100 | 100 | Up to 0/22,4 |
| | | | 200 | |

6.2 Initial (first stage) curing of test specimens

Within (4 ± 1) h of mixing, and without drying out of the specimens, commence the first stage curing of the test specimens in their moulds by sealing each one in a watertight plastic bag or plastic (cling) film. Taking account of the strength gain of the binder (see NOTE 2), keep the test specimens in a controlled environment and for a time as determined at the place of use.

NOTE 1 Placing the test specimens in their moulds in a humidity cabinet is a permitted alternative to using watertight plastic bags.

It is important that all the test specimens commence the freeze thaw cycling at a strength which is a significant proportion, say 80 % to 90 %, of their ultimate strength. Elevated temperature or longer age curing will be required for slow setting and hardening mixtures compared to fast setting and hardening mixtures. 28 days at 20 °C should be appropriate for fast setting mixtures and 28 days at 40 °C or 60 or 90 days at 20 °C has been found appropriate for slow-setting mixtures.

NOTE 2 In the case of fast setting mixtures or mixtures with significant cohesion, it will be possible to remove specimens from their moulds and return them un-moulded to the first stage curing prior to completion of the first stage curing.

6.3 Removal from moulds at the end of first stage curing

Remove the test specimens from the initial curing environment. Remove each test specimen from its mould.

Group the specimens into two sets of three, known as Set A and Set B. Mark each test specimen with a suitable identification code.

NOTE If appropriate, the additional set of three test specimens is known as Set C.

7 Test procedure

7.1 Second stage curing

NOTE Duration of this second stage curing is 2 days for set A and for set C if made. For set B, the duration of second stage curing lasts until completion of the freeze thaw cycling of set A and the subsequent testing for strength of sets A and B.

Place all the sets of test specimens in the second stage curing condition i.e. in the water bath or humidity cabinet or the first stage curing condition, all maintained at (20 ± 2) °C.

In the case of the water bath, adjust the level of the water so that the tops of the specimens are covered to a depth of at least 10 mm for the full period of soaking.

The combined time taken to carry out the procedure described in 6.3 and the placing in the second stage curing condition shall not exceed 60 min.

7.2 Set A — Exposure to freezing after 2 days of second stage curing

Place the electronic thermocouple probe in the can of aggregate and water in the centre of the cooling area. Use the thermocouple output to condition the low temperature cabinet so that the temperature in the can of aggregate is (20 ± 3) °C.

NOTE It may be necessary to stabilise the temperature in the low temperature cabinet before commencement of freeze thaw cycling.

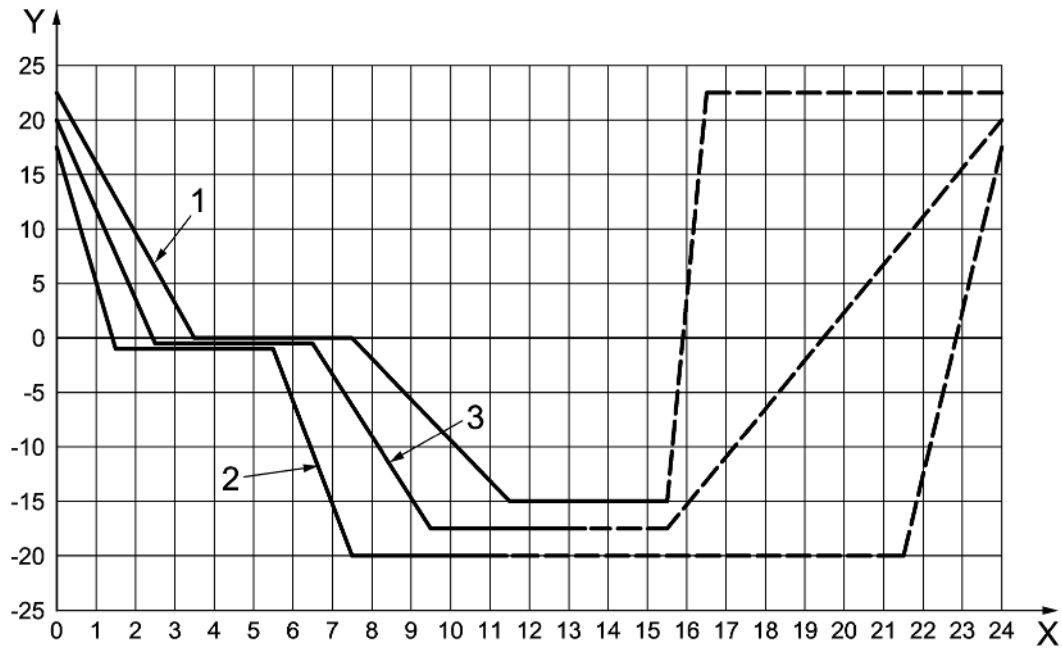
After (48 ± 2) h of second stage curing, remove one of the Set A specimens and quickly remove excess water with a damp cloth. Quickly wrap the saturated test specimen in plastic (cling) film. Do not allow any part of the surface of the test specimen to dry out.

Repeat the removal and wrapping stage with the other two test specimens in Set A.

Place the three wrapped Set A specimens in the cabinet. Ensure that the distance between each test specimens, the can of aggregate and the sidewalls of the cabinet is not less than 50 mm in any direction.

Use the output of the thermocouple in the central can of aggregate to regulate the temperature of the air in the low temperature cabinet over ten freeze thaw cycles. Complete each freeze-thaw cycle within 24 h.

Use a suitable method of control to ensure that the temperature in the central can of aggregate complies with the limits in Figure 1 during each freeze thaw cycle:



Key

- | | | | |
|---|----------------|---|-------------|
| Y | temperature °C | 2 | lower limit |
| X | time in h | 3 | control |
| 1 | upper limit | | |

Figure 1 — Temperature profile for the can of aggregate in the centre of the low temperature cabinet

7.3 Set A — final thawing and conditioning for 24 h prior to strength testing

At the end of the tenth thawing stage, remove the plastic (cling) film from the three test specimens.

Return Set A to the second stage curing condition at (20 ± 2) °C for (24 ± 2) h.

NOTE The second stage curing is where Set B has remained during the exposure of Set A to the freeze thaw cycling.

7.4 Strength testing after final thawing and conditioning

Determine the compressive or tensile strength as appropriate of Set A and B specimens using the method specified in the mixture standard.

The strength determination stage for both sets of test specimens shall be carried out as a continuous operation preventing drying out of the specimens.

If the freeze thaw cycle has been interrupted for any reason (see 7.2), delay the testing of Set B so that the strength of both sets of test specimens is determined at the same time. Report the actual test day in the test report.

NOTE When required, the bulk density of each saturated test specimen can be determined before testing, using the water displacement method specified in EN 12390-7.

The strength of Set C should be determined at the end of second stage curing, using the same method as is used for the other test specimens.

8 Calculation

Calculate

- the mean value of strength R_A for Set A ();
- the mean value of strength R_B for Set B ();
- the freeze thaw retained strength ratio, R_A/R_B .

NOTE The mean strength of Set C can also be compared with the mean strength of Set B using a similar formula.

9 Test report

The test report shall provide the following information.

- a) HBM type, characterisation and relevant part of EN 14227 (e.g. Hydraulic road binder bound mixture 2, 0/20 – G2 – T2 see EN 14227-5);
- b) producer and producer code where applicable and intended source of production;
- c) description of constituents and mixture proportions (% by dry mass);
- d) specimen dimensions, method of specimen manufacture and compactive effort employed for specimen manufacture;
- e) mass and bulk density of specimen at time of test;
- f) curing conditions for first stage curing (e.g. curing temperature, sealed curing, curing in humidity cabinet);
- g) date of commencement of first stage curing;
- h) date of commencement of second stage curing and type of curing employed;
- i) age of specimen and date at commencement of freeze thaw-cycling;
- j) age at time of strength determination after commencement of freeze-thaw cycling and whether any interruptions to freeze-thaw cycling;
- k) freeze-thaw retained strength ratio;
- l) strength after second stage curing if applicable;
- m) change in height in accordance with Annex A if applicable;
- n) state whether test was carried out in accordance with Annex B;
- o) declaration that testing was carried out in accordance with this standard and any deviation from the test method.

Annex A (normative) Change in height

The apparatus used at BAST to measure change in height after freeze thaw is illustrated in the three photographs below.



Figure A.1 — Base of test specimen — 3 balls



Figure A.2 — Top of test specimen — 1 ball



Figure A.3 — Demonstration of height measurement

EN 1367-4 sets out a method for measuring the drying shrinkage of aggregates using concrete prisms. The measurement parts of this method can be used as a basis for this annex. The following text needs some work, but is offered as starting point:

Moulds, of the required dimensions, with a method of fixing three recessed inserts securely to the inside face of the flat base.

A method for fixing the one recessed insert into the top of the test specimen. (This is not the same as the drying shrinkage prisms that are made on their 'side'.)

Stainless steel hemispherical buttons, 8 mm diameter, suitable for screwing into the recessed inserts at each end of the test specimen, once it has been removed from the mould.

Dial gauge, with scale divisions of 0,002 mm and having a maximum error of $\pm 0,002$ mm in any half revolution.

Measuring frame, to rigidly mount the dial gauge, with a recessed end that can be located on the 8 mm diameter hemispherical button in the centre of the top face of the test specimen. The base of the frame has a recessed seating that securely locates the three buttons in the bottom face of the test specimen.

Annex B **(normative)** **Freeze thaw in the presence of salt**

EN 1367-6 is a modification of the EN 1367-1 method for countries where frequent freeze-thaw cycling occurs in the presence of sea water spray or abundant de-icing salt solution.

The boundary conditions are the same as the EN 1367-1 method, except that the water is replaced by 1 % sodium chloride (NaCl) solution.

The NaCl solution is made by mixing 20,0 g of NaCl (analytical grade) with 1 980 g of de-ionized or distilled water.

Variation to method — key points

The use of salt solution means that a humidity cabinet cannot be used for the second stage curing of Set A from day 15 onwards.

Soak the first set of three test specimens (Set A) in NaCl solution for:

- the second stage curing after removal from mould;
- final thawing and conditioning period.

The 'control' set of three test specimens is stored in 'normal' water, not in NaCl solution.

Bibliography

- [1] EN 12390-7, *Testing hardened concrete - Part 7: Density of hardened concrete*
- [2] EN 13286-50, *Unbound and hydraulically bound mixtures - Part 50: Method for the manufacture of test specimens of hydraulically bound mixtures using Proctor equipment or vibrating table compaction*
- [3] EN 13286-51, *Unbound and hydraulically bound mixtures - Part 51: Method for the manufacture of test specimens of hydraulically bound mixtures using vibrating hammer compaction*
- [4] EN 13286-52, *Unbound and hydraulically bound mixtures - Part 52: Method for the manufacture of test specimens of hydraulically bound mixtures using vibrocompression*
- [5] EN 13286-53, *Unbound and hydraulically bound mixtures - Part 53: Methods for the manufacture of test specimens of hydraulically bound mixtures using axial compression*

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