SLOVENSKI STANDARD
SIST EN 1744-5:2007

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Preskusi kemiļ nih lastnosti agregatov ï 5. del: Doloļ evanje kloridnih soli, topnih v kislini

Tests for chemical properties of aggregates - Part 5: Determination of acid soluble chloride salts

Prüfverfahren für chemische Eigenschaften von Gesteinkörnungen - Teil 5: Bestimmung der säurelöslichen Chloride

Essais relatifs aux propriétés chimiques des granulats - Partie 5: Dosage des sels chlorures solubles dans l'acide

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Tests for chemical properties of aggregates - Part 5: Determination of acid soluble chloride salts

Essais pour déterminer les propriétés chimiques des granulats - Partie 5: Détermination de la teneur en chlorures solubles dans l'acide

Prüfverfahren für chemische Eigenschaften von Gesteinkörnungen - Teil 5: Bestimmung der säurelöslichen Chloride

This European Standard was approved by CEN on 28 August 2006.

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.

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Foreword

This document (EN 1744-5:2006) 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of an identical text or by endorsement, at the latest by April 2007, and conflicting national standards shall be withdrawn at the latest by April 2007.

This standard forms part of a series of tests for chemical properties of aggregates. Test methods for other properties of aggregates will be 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 reaction

Part 3: Preparation of eluates by leaching of aggregates

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

Part 6: Determination of the influence of recycled 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, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard specifies the procedure for the determination of acid soluble chloride salts which may be present in aggregates. This test is suitable for aggregates where the chloride content does not derive directly from contact with, or immersion in, saline water. Examples of such aggregates are: recycled aggregates containing hydrated cement, where chloride may be bound as calcium chloroaluminates; and some aggregates from desert areas where chloride is occluded within the aggregate particles.

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 1744-1:1998, *Tests for chemical properties of aggregates — Part 1: Chemical analysis*

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

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

ISO 384, *Laboratory glassware — Principles of design and construction of volumetric glassware*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*

3 Definitions

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

3.1
test portion
sample used as a whole in a single test

3.2
test specimen
when a test method requires more than one determination of a property, the test specimen is the sample used in a single determination

3.3
laboratory sample
reduced sample derived from a bulk sample for laboratory testing

4 Reagents

Unless otherwise stated, use only analytical grade reagents and demineralised water, or water of equivalent purity.

NOTE 1 Unless otherwise stated "%" means "% by mass".

NOTE 2 Where no tolerances are given for reagent volumes or masses, the values quoted are approximate. In such cases volumes delivered from measuring cylinders and indicated masses using the ordinary balance specified in 5.1 are considered sufficiently accurate for the purposes of this European Standard.

NOTE 3 Unless otherwise stated reagent solutions may be assumed to have long-term stability.

NOTE 4 All chemicals should be treated as potential poisons with toxic properties and appropriate precautions taken before their use. Always take time to assess possible hazards before starting any procedures and constant attention should be maintained.

4.1 General requirements for densities

The following concentrated liquid reagents required by this standard shall have the following densities in grams per cubic centimetre at (20 ± 3) °C.

Hydrochloric acid	: 1,18 to 1,19
Nitric acid	: 1,40 to 1,42
Sulfuric acid	: 1,84
Ammonium hydroxide	: 0,88 to 0,91

The degree of dilution shall be indicated as a volumetric sum.

NOTE Ready for use solutions are allowed as an alternative.

4.2 Reagents for determination of acid -soluble chloride salts (Volhard method)

4.2.1 Silver nitrate ($AgNO_3$) solution: 0,100 mol/l is prepared by drying about 20 g of silver nitrate for at least 1 h at a temperature of (110 ± 5) °C, allowing it to cool in a dessicator, weighing (16,987 ± 0,001) g of the dried silver nitrate, then dissolving it in water and diluting it to 1 l in a volumetric flask (5.6). Store the solution in the amber-coloured glass reagent bottle (5.7) and protect from prolonged exposure to sunlight.

4.2.2 Thiocyanate (KSCN or NH_4SCN) solution: approximately 0,1 mol/l is prepared by dissolving 9,7 g of potassium thiocyanate or 7,6 g of ammonium thiocyanate in water and diluting it to 1 l in a volumetric flask.

Pipette 25 ml of silver nitrate solution (4.2.1) into a flask (5.8) and add 5 ml of nitric acid (4.2.3) and 1 ml of ammonium iron (III) sulfate indicator solution (4.2.5).

Add the thiocyanate solution from a burette (5.9) until the first permanent colour change occurs, that is from white opalescence to pale brown. Note the volume of thiocyanate solution (V_0) added.

Calculate the concentration of the thiocyanate solution c_T, (in moles per litre), from the following equation:

$$c_T = 2{,}5/V_0 \qquad (1)$$

where V_0 is the volume of thiocyanate added (in millilitres).

Standardize the solution at weekly intervals or before use if the tests are carried out infrequently.

4.2.3 Nitric acid (HNO_3): approximately 6 mol/l is prepared by adding 100 ml of nitric acid (4.1) to 150 ml of water, boiling the diluted acid in a fume cupboard (5.11) until it is colourless and allowing to cool to room temperature.

4.2.4 Chloride free technical grade 3,5,5,-trimethylhexan-1-ol.

4.2.5 Ammonium iron (III) sulfate $NH_4Fe(SO_4)_2.12H_2O$ indicator solution is prepared by adding 60 g of water to 50 g of ammonium iron (III) sulfate, warming until it dissolves, and adding 10 ml of nitric acid (4.2.3).

Allow the solution to cool to room temperature and store it in a glass bottle (5.10).

5 Apparatus

All apparatus shall comply with the general requirements of EN 932-5.

All volumetric glassware shall be of class B accuracy as defined in ISO 384. Volumetric glassware of class A shall be used for audit test and type test.

5.1 Balance, capable of weighing up to 100 g, readable to the nearest 0,1 mg.

5.2 Two filter funnels, of approximately 100 mm diameter with coarse filter papers (pore diameter approximately 20 µm); of diameters appropriate to the size of the funnels.

5.3 Two 250 ml beakers.

5.4 Glass rods.

5.5 Pipette: 1 ml, 5 ml, 25 ml

5.6 Two volumetric flasks, capacity of 1 l, complying with the requirements of ISO 1042.

5.7 Amber-coloured glass reagent bottles.

5.8 Stoppered conical flasks, 100 ml and 250 ml capacity.

5.9 Burette, 50 ml size, graduated to 0,1 ml.

5.10 Plain glass reagent bottles.

5.11 Fume cupboard.

6 Principle

This method gives the total halide content except for fluoride and expresses the result as chloride ion content.

The aggregate sample is reduced to a fine powder and extracted with boiling dilute nitric acid.

NOTE Sulfides are oxidised into sulfates and do not interfere.

The dissolved chloride is precipitated using a known volume of standard silver nitrate solution. After boiling, the precipitate and the remaining solid aggregate are washed with a dilute nitric acid and discarded. The filtrate and washings are cooled to room temperature and the residual silver nitrate is titrated with a standard thiocyanate solution using an iron (III) salt as an indicator (Volhard method).

7 Sampling

The laboratory sample shall be taken in accordance with the procedures specified in EN 932-1. Ensure that the laboratory sample is representative of the moisture content as well as the solids.

8 Preparation of test portion

The test specimen is prepared using the procedure given in EN 1744-1:1998, 12.3. Take approximately 2 g of the powdered sample as the test specimen.

9 Procedure

Weigh the test specimen to the nearest 0,1 mg and record it as “m”; place it in a 250 ml beaker, add 50 ml of water and while stirring with a glass rod, 50 ml of dilute nitric acid (1 + 2). Heat the mixture to boiling, stirring occasionally, and boil for 1 min.

NOTE Aggregates containing significant amounts of carbonates will froth on addition of the acid. In these cases, add the acid slowly while continuously stirring.

Add 5 ml of the 0,100 mol/l silver nitrate solution by pipette (5.5) into the boiling solution. Then boil for a maximum of 1 min and filter through a filter paper (5.2) washed before use with dilute nitric acid (1 + 100) into a 500 ml flask (5.6). Wash the beaker, glass rod and filter with dilute nitric acid (1 + 100) until the volume of filtrate and washings is 200 ml. Cool the filtrate and washings to room temperature.

Add 5 ml ammonium iron (III) sulfate indicator solution and titrate with the thiocyanate solution, shaking vigorously until a drop of this solution produces a faint brown coloration which no longer disappears on shaking.

Note the volume V_1 of the thiocyanate solution added.

If the chloride content of the aggregate exceeds 0,85 %, it will be necessary to start the test again with a smaller test portion.

Repeat the procedure with a second test specimen.

10 Calculation and expression of results

Calculate the chloride content (in %) from the following equation:

$$C_a = 3{,}545 \times (5\text{-}10\ C_T\ V_1)/1000 \times 100/m = 0{,}3545(5\text{-}10\ C_T\ V_1)/m \quad (2)$$

where:

m is the mass of the test portion (in grams);

V_1 is the volume of thiocyanate solution used for the titration of the test solution (in millilitres);

C_T is the concentration of the thiocyanate solution (in moles/litre, see 4.2.2).

The result of the test is given as the mean of the determinations on the two test specimens, and recorded to the nearest 0,01 %.

11 Test report

a) reference to this European Standard;

b) source of the sample;

c) designation of the sample;

d) mass of dry sample tested, (in grams);

e) result of the test;

f) date of the test.