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Preskusi kemičnih lastnosti agregatov - 1. del: Kemijska analiza

Tests for chemical properties of aggregates - Part 1: Chemical analysis

Prüfverfahren für chemische Eigenschaften von Gesteinskörnungen - Teil 1: Chemische Analyse

Essais visant à déterminer les propriétés chimiques des granulats - Partie 1: Analyse chimique

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This draft amendment is submitted to CEN members for unique acceptance procedure. It has been drawn up by the Technical Committee CEN/TC 154.

This draft amendment A1, if approved, will modify the European Standard EN 1744-1:2009. If this draft becomes an amendment, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for inclusion of this amendment into the relevant national standard without any alteration.

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

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EN 1744-1:2009/FprA1:2012 (E)

Foreword

This document (EN 1744-1:2009/FprA1:2012) has been prepared by Technical Committee CEN/TC 154 “Aggregates”, the secretariat of which is held by BSI.

This document is currently submitted to the Unique Acceptance Procedure.

1 Modification to 11.1, Determination of total sulfur content by acid digestion (Reference method)

Replace the existing text in 11.1 with the following:

"

11.1.1 Principle

An aggregate test specimen is treated with hydrogen peroxide and hydrochloric acid to convert any sulfur compounds present to sulfates. Any sulfates are precipitated as barium sulfate (BaSO_4) and weighed. The sulfur content is expressed as a percentage by mass of the aggregate.

11.1.2 Sampling

The laboratory sample shall be taken in accordance with the procedures specified in EN 932-1.

The laboratory sample should be representative of the moisture content of the batch.

11.1.3 Preparation of test portion

Reduce the laboratory sample by the procedures specified in EN 932-2 to an amount not less than the mass specified in Table 1 appropriate to the upper (*D*) sieve size of the aggregate.

If necessary, dry the sample at a temperature of (110 ± 5) °C.

NOTE Drying at a higher temperature will oxidise any sulfides.

Stepwise crush and reduce the sub-sample to a mass of approximately 20 g and then grind the particles until they all pass the 125 μm sieve. Take approximately 1 g of this material as the test specimen.

11.1.4 Procedure

Weigh the test portion to the nearest 0,1 mg (m_4) into a 400 ml high form beaker (5.2.9). Add 20 ml distilled water to elutriate, using a glass rod and watch glass. In the fume cupboard, add 10 ml of hydrogen peroxide (4.6.1) and warm for 30 min below the boiling point.

After the dissolution, add 20 ml hydrochloric acid (1+1) (4.1) and leave the test specimen in a heat bath for 30 min.

Add a small amount of filter paper pulp and warm up the content of the beaker to below the boiling point.

Make the solution alkaline by adding ammonium hydroxide (4.1). Check the alkalinity by using methyl red as an indicator (4.6.2), or by using a pH-meter (5.2.8). Simmer for 30 s and then filter under gentle suction using a medium porosity filter paper. Wash the contents of the filter once with a little hot, demineralized water and reserve the filtrates.

Transfer the paper filter and the particles retained on it into the beaker and dissolve in 5 ml concentrated hydrochloric acid (4.1) to which has been added 70 ml hot water.

Repeat the procedure above (boil, precipitate, filter and wash) and reject the precipitate if any. Acidify the combined filtrates and washings (which should be about 220 ml in all) with 1 ml concentrated hydrochloric acid and bring it to the boil. Boil for $(5 \pm 0,5)$ min.

While maintaining the solution at boiling point and stirring vigorously, add drop-by-drop 10 ml of the barium chloride solution (4.5.2) heated to just below boiling.

Mature, filter and ignite the barium sulfate precipitate as specified in 10.1.5.

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Weigh to the nearest 0,1 mg and calculate the mass of precipitate m_5 .

11.1.5 Calculation and expression of results

Calculate the total sulfur content of the aggregate, expressed as S to the nearest 0,1%, from the following formula:

$$S = m_5/m_4 \cdot 13,74 \text{ (in \%)} \quad (11)$$

where

m_5 is the mass of precipitate in grams;

m_4 is the mass of the test portion in grams.

NOTE A statement on the precision of the determination of total sulfur content is given in Annex A."

2 Modification to Clause 12, Determination of acid soluble sulfates

Replace the existing text in Clause 12 with the following:

"

12.1 Principle

Sulfates are extracted from a test portion of the aggregate by dilute hydrochloric acid. The sulfate ion content is determined by gravimetry and expressed as a percentage by mass of the aggregate.

12.2 Sampling

The laboratory sample shall be taken in accordance with the procedures specified in EN 932-1.

The laboratory sample should be representative of the moisture content of the batch.

12.3 Preparation of test portion

Reduce the laboratory sample by the procedures specified in EN 932-2 to an amount not less than the mass specified in Table 1 appropriate to the upper (*D*) sieve size of the aggregate.

If necessary, dry the sample at a temperature of (110 ± 5) °C.

NOTE Drying at a higher temperature will oxidise any sulfides.

Stepwise crush and reduce the sub-sample to a mass of approximately 20 g and then grind the particles until they all pass the 125 µm sieve. Take approximately 8 g of this material as the test portion.

12.4 Procedure

Weigh the test portion to the nearest 0,1 mg (m_6). Place 360 ml water and 40 ml of concentrated hydrochloric acid (4.1) in a 1000 ml conical flask (5.2.9) and heat to boiling point. Remove from the source of heat and, while stirring, sprinkle the test portion on to the acid solution. Break up the solids with the flattened end of a glass-stirring rod. Allow the solution to digest for (15 ± 1) min at a temperature just below boiling.

Filter the residue through a medium filter paper into a 1000 ml beaker. Thoroughly wash the contents of the filter with hot demineralized water. Check the washings to ensure the absence of chloride ions using the silver nitrate test (6.6)

Adjust the volume to about 600 ml with demineralized water and warm up the content of the beaker to below the boiling point.

Make the solution alkaline by adding ammonium hydroxide solution (4.1.2). Check the alkalinity by using methyl red as an indicator (4.6.2), or by using a pH-meter (5.2.8). Simmer for 30 s and then filter under gentle suction using a medium porosity filter paper. Wash the contents of the filter at least 3 times with a little hot, demineralized water and reserve the filtrates. Acidify by means of hydrochloric acid (1+11) to red coloration of methyl red indicator (4.6.2)

Bring to the boil and boil for $(5 \pm 0,5)$ min and then check that the solution is clear. If it is not clear, start the test again using a new test portion.

While maintaining the solution at boiling point and stirring vigorously, slowly add 40 ml of the barium chloride solution (4.5.2) heated to just below boiling. Mature, filter and ignite the barium sulfate precipitate as specified in 10.1.5.

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Weigh to the nearest 0,1 mg and calculate the mass of precipitate (m_7).

12.5 Calculation and expression of results

Calculate the acid soluble sulfate content of the aggregate, expressed as SO_3 to the nearest 0,1%, from the following formula:

$$\text{Sulfate content } (\text{SO}_3) = m_7/m_6 \cdot 34,30 \text{ (in \%)} \quad (12)$$

where

m_7 is the mass of precipitate in grams;

m_6 is the mass of the test portion in grams.

NOTE 1 Results can be expressed in SO_4 using the following formula:

$$\text{Sulfate content } (\text{SO}_4) = m_7/m_6 \cdot 41,16 \text{ (in \%)}$$

NOTE 2 A statement on the precision of the determination of the acid soluble sulfate content is given in Annex A."

3 Modification to Clause 17, Determination of loss on ignition

Replace the existing text in Clause 17 with the following:

"

17.1 Principle

The loss on ignition is determined in an oxidizing atmosphere. By igniting the aggregate in air at (950 ± 25) °C the carbon dioxide and the water not evaporated during drying are driven off, as are any oxidizable volatile elements present.

NOTE 1 If the aggregate contains non-volatile oxidizable constituents, as in the case of blast-furnace slags, the loss on ignition should be corrected in accordance with EN 196-2:2005, 7.4.

NOTE 2 For aggregates produced from incinerator residues, the ignition temperature should be (480 ± 25) °C.

NOTE 3 This test may be used as an alternative test for humus content. In that case temperature should be (480 ± 25) °C.

17.2 Sampling and preparation of test portion

The laboratory sample shall be taken in accordance with the procedures specified in EN 932-1.

The laboratory sample should be representative of the moisture content of the batch.

Reduce the laboratory sample by the procedures specified in EN 932-2 to an amount not less than the mass specified in Table 1 appropriate to the upper (*D*) sieve size of the aggregate.

If necessary, dry the sample at a temperature of (110 ± 5) °C.

NOTE Drying at a higher temperature will oxidise any sulfides.

Stepwise crush and reduce the sub-sample to a mass of approximately 20 g and then grind the particles until they all pass the 125 µm sieve. Take $(1 \pm 0,05)$ g of this material as the test specimen.

17.3 Procedure for the determination of loss on ignition

Weigh the test portion to the nearest 0,1 mg (m_{13}) into a crucible (5.6.2) which has been previously ignited and tarred. Place the crucible in the electric furnace (5.2.2) controlled at (950 ± 25) °C. Leave the crucible in the furnace for at least 60 min. Cool the crucible to room temperature in a desiccator (5.2.16) and then re-weigh (m_{14}).

In the case of calcareous aggregates, heating to 950 °C should proceed slowly to minimize the risk of violent decrepitation.

17.4 Calculation and expression of results

Calculate the loss on ignition of the aggregate from the following formula:

$$\text{Loss on ignition} = \frac{m_{13} - m_{14}}{m_{13}} \cdot 100 \text{ (in \%)} \quad (18)$$

where

m_{13} is the mass of the initial test portion in grams;