RATIFIED EUROPEAN TEXT

EN1744-1:1998

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This ratified text was approved on

1998-02-15

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 1744-1

March 1998

ICS 71.040.40; 91.100.20

Descriptors: aggregates, chemical properties, chemical analysis, determination, chlorides, sulphates, sulphur, sulphites, organic compounds, lime, solubility, ignition losses

English version

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

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

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

This European Standard was approved by CEN on 15 February 1998.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 1744-1:1998 E

Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 154 " Aggregates", the secretariat of which is held by BSI.

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 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

The other parts of EN 1744 will be:

Part 2	Determination of resistance to alkali reaction
Part 3	Water leaching test
Part 4	Determination of water susceptibility of fillers for bituminous mixtures

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 September 1998, and conflicting national standards shall be withdrawn at the latest by December 1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies procedures for the chemical analysis of aggregates. It specifies the reference procedures and, in certain cases, an alternative method which can be considered as giving equivalent results.

If other methods are used it is necessary to show that they give results equivalent to those given by the reference methods.

NOTE: In cases of dispute, only the reference procedures should be used.

Unless otherwise stated, the test methods specified in this European Standard may be used for factory production control, for audit tests or for type tests.

2 Normative references

This European Standard incorporates by dated or by undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies.

EN 196-1:1993	Methods of testing cement Part 1: Determination of strength
EN 196-2:1987	Methods of testing cement Part 2: Chemical analysis of cement
EN 196-3:1987	Methods of testing cement Part 3: Determination of setting time and soundness
ENV 197-1:1992	Cement - composition, specifications and conformity criteria. Part 1: Common cements
EN 932-1:1996	Tests for general properties of aggregates Part 1: Methods for sampling
prEN 932-2	Tests for general properties of aggregates Part 2: Methods for reducing laboratory samples
prEN 932-5	Tests for general properties of aggregates Part 5: Common equipment and calibration
EN 933-2:1995	Tests for geometrical properties of aggregates Part 2: Determination of particle size distribution - Test sieves, nominal size of apertures
prEN 1015-4	Methods of test for mortar for masonry Part 4: Determination of consistence of fresh mortar (by plunger penetration)

prEN 1015-9	Methods of test for mortar for masonry Part 9: Determination of service life of fresh mortars
prEN 1015-11	Methods of test for mortar for masonry Part 11: Determination of flexural and compressive strength of hardened mortar
prEN 1097-6	Tests for mechanical and physical properties of aggregates Part 6: Determination of particle density and water absorption
ISO 384:1978	Laboratory glassware - Principles of design and construction of volumetric glassware.
ISO 648:1977	Laboratory glassware - One-mark pipettes
ISO 650:1977	Relative density 60/60 degrees F hydrometers for general purposes
ISO 1042:1983	Laboratory glassware - One-mark volumetric flasks

ISO 4788: 1980 Laboratory glassware - Graduated measuring cylinders

3 Definitions

For the purposes of this standard, the following definitions apply:

3.1 test portion: The 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: A reduced sample derived from a bulk sample for laboratory testing.

3.4 constant mass: Successive weighings after drying at least 1 h apart not differing by more than 0,1 %.

NOTE: 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.

3.5 batch: A production quantity, a delivery quantity, a partial delivery quantity (railway wagon-load, lorry-load, ship's cargo) or a stockpile produced at one time under conditions that are presumed uniform.

NOTE: With a continuous process the quantity produced during an agreed period is treated as a batch.

3.6 fines: The particle size fraction of an aggregate which passes the 0,063 mm sieve.

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 balances specified in **5.2.4** and **5.2.5** 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 °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 1: For example in **4.11.4**, "hydrochloric acid (1+1)" means that 1 volume of concentrated hydrochloric acid is to be mixed with 1 volume of water.

NOTE 2: Ready for use solutions are allowed as an alternative.

4.2 Reagents for determination of water-soluble chloride salts using the Volhard method (clause 7).

4.2.1 Silver nitrate ($AgNO_3$) solution, 0,100 mol/l, prepared by drying about 20 g of silver nitrate for at least 1 h at a temperature of (110 ± 5) °C, allowing to cool in a desiccator and then weighing (16,987 ± 0,001) g of the dried silver nitrate, dissolving in water and diluting to 1 l in a volumetric flask (**5.3.6**). Store the solution in the amber-coloured glass reagent bottle (**5.2.14**) and protect from prolonged exposure to sunlight.

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

Pipette 25 ml of silver nitrate solution (**4.2.1**) into a flask (**5.3.5**) 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 (**5.2.13**) until the first permanent colour change occurs, that is from white opalescence to pale brown. Note the volume of thiocyanate solution added.

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

$$c_T = 2{,}5/V_1$$

where:

V_1 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, prepared by adding 100 ml of nitric acid (**4.1**) to 150 ml water, boiling the diluted acid in a fume cupboard (**5.2.17**) until it is colourless and allow 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, prepared by adding 60 g of water to 50 g ammonium iron (III) sulfate, warming to dissolve, and adding 10 ml nitric acid (**4.2.3**).

Allow the solution to cool to room temperature and store in a glass bottle (**5.2.15**).

4.3 Reagents for potentiometric determination of water-soluble chloride salts (clause 8)

4.3.1 Silver nitrate ($AgNO_3$) solution, 0,01 mol/l, prepared using the same procedure as specified in **4.2.1**, but dissolving 1,699 g of dried silver nitrate in a 1 l volumetric flask (**5.3.6**).

4.3.2 Sodium chloride (NaCl) solution, 0,02 mol/l, prepared by drying about 2 g of sodium chloride at a temperature of (110 ± 5) °C for 1 h to 2 h, allowing to cool and then weighing (1,169 ± 0,001) g of the dried sodium chloride, dissolving in water and diluting to 1 l in a volumetric flask (**5.3.6**).

4.4 Reagent for factory production control determination of water-soluble chloride salts using the Mohr method (clause 9)

Potassium chromate (K_2CrO_4) solution, prepared by dissolving 10 g of potassium chromate in 100 ml water.

4.5 Reagents for determination of water-soluble sulfates (clause 10)

4.5.1 Hydrochloric acid (HCl) solution, made by adding 200 ml concentrated hydrochloric acid (relative density 1,18) to 800 ml water.

4.5.2 Barium chloride ($BaCl_2$) solution, made by dissolving 100 g of barium chloride ($BaCl_2.2H_2O$) in 1 l of water, and filtered through a medium grade filter paper before use.

4.6 Reagents for determination of total sulfur content (clause 11)

4.6.1 Bromine.

4.6.2 Indicator methyl red (dissolve 20 mg methyl red powder in 50 ml of ethanol, then add 50 ml of water).

4.7 Reagents for determination of sulfide content (clause 13)

4.7.1 Lead acetate solution (dissolve approximately 0,2 g of lead acetate $\{Pb(CH_3COO)_2.3H_2O\}$ in water and make up to 100 ml).

4.7.2 Ammoniacal zinc sulfate solution (dissolve 50 g of zinc sulfate ($ZnSO_4.7H_2O$) in 150 ml water and add 350 ml of concentrated ammonium hydroxide (NH_4OH)). Leave to stand for at least 24 h and filter through a medium grade filter paper.

4.7.3 Tin (II) chloride ($SnCl_2.2H_2O$).

4.7.4 Metallic chromium Cr powder form (**CAUTION : CARCINOGENIC**).

4.7.5 Standard potassium iodate solution containing 0,0167 mol/l; dissolve successively in freshly boiled and cooled water in a 1 l volumetric flask, (3,6 ± 0,1) g to the nearest 0,1 mg (**6.3**) of potassium iodate KIO_3, dried at (110 ± 5) °C, two pellets (about 0,4 g) of sodium hydroxide (NaOH) and 25 g of potassium iodide (KI).

Make up to the mark with freshly boiled and cooled water.

NOTE 1 : A trace of sodium hydroxide stabilises this solution for a considerable time; the solution should be discarded when it becomes discoloured.

The factor F of this solution is calculated from the following equation:

$$F = \frac{m_1}{3{,}5668}$$

where:

m_1 is the mass of the portion of potassium iodate.

NOTE 2 : If the sulfide content is low (less than 0,1 %), solutions ten times less concentrated should be used. They are prepared by pipetting 100 ml of the solutions (**4.7.5** and **4.7.6**) into 1 l volumetric flasks and making up to the mark with water.

4.7.6 Sodium thiosulfate solution approximately 0,1 mol/l.

Dissolve 24,82 g of sodium thiosulfate ($Na_2S_2O_3.5H_2O$) in water and make up to 1 l. Before each test series, determine the factor f of this solution as specified as follows.

Standardize the thiosulfate solution in one of the following ways.

a) Standardization carried out preferably in relation to the standard potassium iodate solution (**4.7.5**).

For this standardization, pipette 20 ml of the standard potassium iodate solution into a 500 ml conical flask and dilute with approximately 150 ml of water. Acidify with 25 ml of hydrochloric

acid (1 + 1) and titrate with the approximately 0,1 mol/l sodium thiosulfate solution to a pale yellow colour.

Then add 2 ml of the starch solution (**4.7.7**) and continue the titration until the colour changes from blue to colourless.

The factor *f* of this solution is calculated from the following equation:

$$f = \frac{20 \times 0{,}01667 \times 214{,}01 \times F}{3{,}5668 \times V_2} = 20 \frac{F}{V_2}$$

where:

F is the factor of the standard potassium iodate solution (**4.7.5**) expressed in moles per litre;

V_2 is the volume of the approximately 0,1 mol/l sodium thiosulfate solution used for the titration;

3,5668 g/l of potassium iodate corresponds to a solution with exactly 0,01667 mol/l of potassium iodate;

214,01 is the molecular mass of KIO_3.

b) Standardization carried out in relation to a known quantity of potassium iodate.

For this standardization, place in a 500 ml conical flask (70 ± 5) mg of potassium iodate and dissolve in approximately 150 ml of water.

Add about 1 g of potassium iodide, acidify with 25 ml of hydrochloric acid (1 + 1) and titrate with the approximately 0,1 mol/l sodium thiosulfate solution until a pale yellow colour is obtained. Then add 2 ml of the starch solution (**4.7.7**) and titrate until the colour changes from blue to colourless.

The factor *f* of this solution is calculated from the following equation:

$$f = \frac{1\,000 \times m_2}{3{,}5668 \times V_3} = 280{,}3634 \frac{m_2}{V_3}$$

where:

m_2 is the mass of the portion of potassium iodate in grams;
V_3 is the volume of the approximately 0,1 mol/l sodium thiosulfate solution used for the titration;
3,5668 g/l of potassium iodate corresponds to a solution with exactly 0,01667 mol/l of potassium iodate.

4.7.7 Starch solution (to 1 g of starch (water soluble), add 1 g of potassium iodide KI, dissolve in water and make up to 100 ml).

4.8 Reagents for determination of lightweight contaminators (see 14.2)

4.8.1 Zinc chloride solution, obtained by dissolving 7 kg of $ZnCl_2$ in 3 l of water in order to obtain a saturated solution of density of (1,98 ± 0,02) g/cm^3 at (20 ± 3) °C. The relative density of the solution, after cooling to room temperature, shall be checked using a suitable hydrometer (**5.8.3**).

NOTE: Zinc chloride solution is moderately irritating to skin and mucous membranes.

4.8.2 Sodium polytungstate solution (as an alternative to **4.8.1**), prepared by dissolving $3Na_2WO_4.9W0_3.H_2O$ crystals in water until the density of well stirred solution containing no undissolved crystals is (1,98 ± 0,02) g/cm^3 at (20 ± 3) °C.

4.9 Reagents for determination of humus content (see 15.1)

4.9.1 Sodium hydroxide solution, a 3 % NaOH solution, obtained by dissolving 30 g of sodium hydroxide pellets in water, cooling to room temperature and diluting to 1 l in a volumetric flask.

4.9.2 Standard colour solution, prepared by dissolving 45,0 g $FeCl_3.6H_2O$ and 5,50 g $CoCl_2.6H_2O$ in 279,5 g of water with 1 ml concentrated HCl. This solution is stored in a glass bottle and is stable for at least 2 weeks.

4.10 Reagents for determination of fulvo acid content (see 15.2)

4.10.1 Hydrochloric acid (1 + 23).

4.10.2 Tin (II) chloride solution. Dissolve 22,5 g $SnCl_2.2H_2O$ in 1 l of hydrochloric acid (**4.10.1**). The quality of this solution will be retained for 2 weeks.

4.11 Reagents for complexometric determination of free lime (see 18.1)

4.11.1 Ethanediol (Ethylene glycol), fresh, anhydrous.

4.11.2 Propan-2-ol (Isopropanol), anhydrous.

4.11.3 Filter paper pulp, in anhydrous ethanediol.

4.11.4 Hydrochloric acid diluted (1+1).

4.11.5 Triethanolamine.

4.11.6 m-Nitrophenol (0,1 g in 100 ml H_20).

4.11.7 Sodium hydroxide solution 2 mol/l, obtained by dissolving 80 g of sodium hydroxide pellets in water, cooling to room temperature and diluting to 1 l in a volumetric flask.

4.11.8 Indicator, grind together with pestle and mortar 1 g of murexide (ammonium purpurate) and 100 g NaCl.

4.11.9 EDTA solution 1/112 mol/l, (3,3 ± 0,1) g of ethylenediaminetetra - acetic acid disodium salt, dried to constant mass at 80 °C, dissolved in water and made up to 1 l. This solution needs to be standardized against a solution of known calcium content (**4.11.10**).

4.11.10 Standard calcium solution (1 ml = 1 mg of calcium oxide). Dissolve (1,785 ± 0,001) g of pure calcium carbonate (**4.11.11**) dried at (110 ± 5) °C in a slight excess of (1 + 4) hydrochloric acid. Boil the solution to expel carbon dioxide, cover and cool to room temperature and dilute to 1 l with water in a volumetric flask (**5.3.6**).

NOTE: Commercially standardized solutions are available, e.g. (1,000 ± 0,002) g CaO/l.

4.11.11 Calcium carbonate ($CaCO_3$) precipitated grade, volumetric standard.

4.11.12 Soda-lime, granulated.

4.12 Reagent for conductimetric determination of free lime (see 18.2)

4.12.1 Ethanediol (**4.11.1**).

4.13 Reagents for acidimetric determination of free lime (see 18.3)

4.13.1 Ethyl acetoacetate, anhydrous grade.

4.13.2 2-methyl propan-l-ol, (isobutyl alcohol) anhydrous grade.

4.13.3 Thymol blue indicator (thymolsulfonephthalein).

4.13.4 Hydrochloric acid, (**4.1**).

4.13.5 Solvent solution, 450 ml of ethyl acetoacetate in 3 l of 2-methylpropan-l-ol.

4.13.6 Indicator (0,1 g of thymol blue indicator powder dissolved in 100 ml of 2-methylpropan-l-ol).

4.13.7 Hydrochloric acid solution approximatively 0,2 mol/l.

To prepare this solution, make up 17 ml of hydrochloric acid (**4.1**) to 1 l with 2-methylpropan-l-ol.

To standardize this solution, weigh (100 ± 0,1) mg of calcium carbonate (**4.11.11**) in a crucible (**5.6.2**) and calcine for 1 h at 1000 °C. Extract the free lime and titrate in accordance with **18.3.3**.

Determine the *k* factor from the following equation:

$$k = \frac{56{,}08}{100{,}09} \times \frac{100}{V_4}$$

where:

V_4 is the volume of hydrochloric acid added (in millilitres);
k represents the number of milligrams of free CaO per millilitre of standardized hydrochloric acid solution.

4.13.8 Sodium hydroxide on support granulated about 0,8 mm to 1,6 mm for elementary analysis.

4.14 Reagent for the determination of the expansion of steel slag (see 19.3)

4.14.1 Silicone oil.

4.14.2 Hydrochloric acid diluted (1 + 5).

5 Apparatus

5.1 General requirements

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

Unless otherwise stated, all volumetric glassware shall be of class B accuracy as defined in ISO 384. Volumetric glassware of class A accuracy shall be used for audit tests and for type tests.

NOTE: Where no tolerances are specified for dimensions, the values quoted are approximate.

5.2 Apparatus for general purposes

5.2.1 Well ventilated oven, capable of being controlled to maintain a constant temperature in the range of 40 °C to 150 °C with a precision of ± 5 °C, equipped with a tray of non-corrodible material.

5.2.2 Electric muffle furnace capable of being controlled to maintain a constant temperature in the range of 800 °C to 1100 °C with a precision of ± 25 °C.

5.2.3 Crushing and grinding equipment to reduce aggregates to sizes so that they pass through sieves suitable for particular tests while producing a minimum of fines.

5.2.4 Balance, capable of weighing up to 10 kg, readable to the nearest 1 g.

5.2.5 Balance, capable of weighing up to 1 kg, readable to the nearest 0,01 g.

5.2.6 Analytical balance, capable of weighing up to 100 g, readable to the nearest 0,1 mg.

5.2.7 Hot plate with magnetic stirrer.

5.2.8 pH meter, readable to 0,1 pH units.

5.2.9 Beakers, conical flasks, funnels and filter paper.

5.2.10 Pipettes, 25 ml, 50 ml and 100 ml, complying with the requirements of ISO 648.

5.2.11 Graduated measuring cylinders, capacity 10 ml, 250 ml and 500 ml, complying with the requirements of ISO 4788.

5.2.12 Wash bottles, containing demineralised water.

5.2.13 Two burettes, 50 ml size, graduated to 0,1 ml.

5.2.14 Amber-coloured glass reagent bottles.

5.2.15 Plain glass reagent bottles.

5.2.16 Desiccators.

5.2.17 Fume cupboard.

5.3 Additional apparatus required for determination of water-soluble chloride salts following Volhard (see clause 7)

5.3.1 Test sieve, 16 mm square hole perforated plate complying with the requirements of EN 933-2.

5.3.2 Two glass, plastic or metal bottles, wide-mouthed, with well fitting stoppers.

NOTE: The bottles should be approximatively 5 l capacity when testing coarse aggregates or lightweight aggregates or 2 l when testing fine aggregates.

5.3.3 Mechanical shaker or roller, to take the extraction bottles (**5.3.2**).

5.3.4 Two filter funnels, of approximately 100 mm diameter with medium and fine grade filter papers of a diameter appropriate to the size of the funnel.

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

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

5.4 Additional apparatus required for potentiometric determination of water-soluble chloride salts (see clause 8)

5.4.1 A potentiometric titrator suitable for the determination of chloride ion concentration with an electrode system consisting of:

a) measuring electrode - either a silver electrode (preferably chloridised) or a chloride ion - selective electrode.

b) reference electrode - either mercurous sulfate or a double junction silver/silver chloride with chloride free electrolyte in the outer chamber.

5.5 Additional apparatus required for factory production control determination of water soluble chloride salts following Mohr (see clause 9)

5.5.1 Two wide-mouthed plastic bottles of 1 l, with stoppers.

5.6 Additional apparatus required for determination of water-soluble sulfates (see clause 10)

5.6.1 Sintered silica filtering crucibles, porosity grade 4, approximately 35 mm in diameter and 40 mm in height.

5.6.2 Ignition crucibles, as alternative to **5.6.1**, approximately 35 mm in diameter and 40 mm in height and capable of maintaining a constant mass when heated to 1100 °C.

NOTE: Porcelain, silica or platinum are suitable materials for ignition crucibles.

5.7 Additional apparatus required for determination of sulfide content (see clause 13)

5.7.1 Typical apparatus for the determination of sulfide content is shown in figure 1.

5.8 Additional apparatus required for determination of lightweight contaminators (see 14.2)

5.8.1 300 µm and 250 µm sieves complying with EN 933-2.

5.8.2 Porcelain evaporating basin.

5.8.3 Hydrometer, range 1,950 to 2,000, complying with the requirements of ISO 650.

5.9 Additional apparatus required for determination of humus content (see 15.1)

5.9.1 4 mm sieve, complying with EN 933-2.

5.9.2 A clear, cylindrical glass bottle with stopper. The capacity of the bottle shall be approximately 450 ml and the external diameter approximately 70 mm.

5.10 Additional apparatus required for determination of fulvo acid content (see 15.2)

5.10.1 Glass stirring rod.

5.10.2 Glass filter funnel.

5.10.3 180 mm diameter medium grade filter paper.

5.10.4 Hot plate.

5.10.5 Standard colour plates (A to G)[1].

5.11 Additional apparatus required for determination of organic contaminators by mortar method (see 15.3)

5.11.1 Stop-watch or a timer, readable to 1 s.

5.11.2 Refractory porcelain or silica basins of size suitable for placing inside the muffle furnace.

5.11.3 Plunger test apparatus conforming with the requirements of prEN 1015-4.

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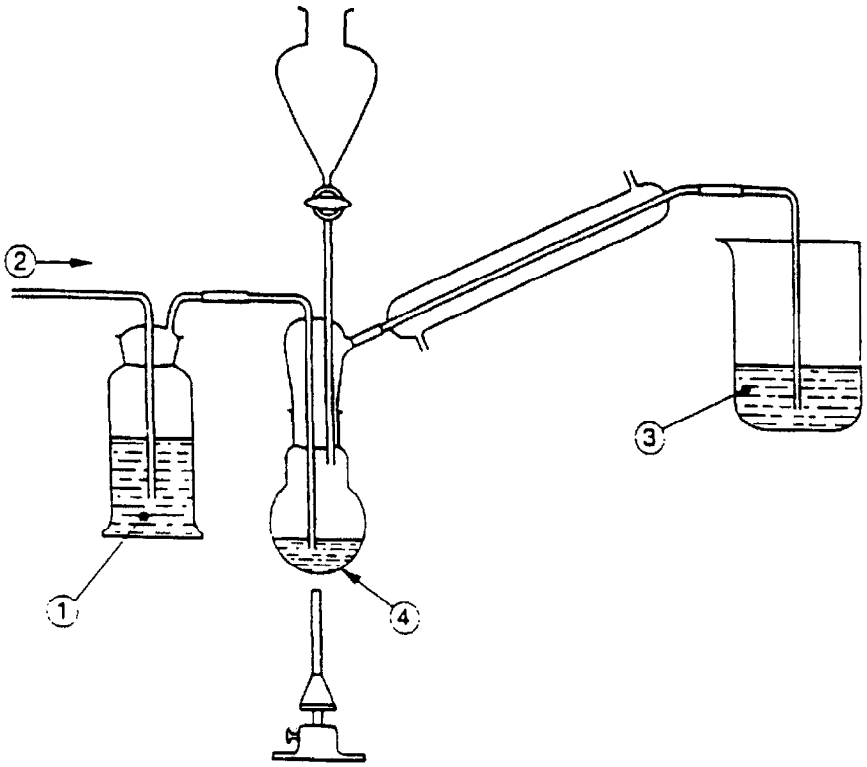
1) Obtainable from the Publication Department of the Dutch Association of Cement Industry
Sint Teunislaan 1-5231 BS, 's Hertogenbosch,
The Netherlands



1 Lead acetate solution (**4.7.1**)
2 Nitrogen or argon
3 Ammoniacal solution of zinc sulfate (**4.7.2**)
4 Reaction flask

Figure 1: Example of apparatus for the determination of sulfide

5.11.4 Mixer complying with the requirements of EN 196-1.

5.11.5 Stiffening rate apparatus conforming with prEN 1015-9.

5.11.6 Flexural and compressive strength apparatus conforming to the requirements of prEN 1015-11.

5.11.7 Electric muffle furnace, with a capacity allowing calcination of a 2 kg aggregate portion, capable of maintaining a temperature of (480 ± 25) °C.

5.12 Additional apparatus required for complexometric determination of free lime (see 18.1)

5.12.1 Erlenmeyer flask, 250 ml capacity, with ground glass stopper.

5.12.2 Volumetric flask, 500 ml capacity.

5.12.3 Magnetic stirrer with temperature controlled water bath.

5.12.4 Sintered glass filter, 10 mm to 16 mm diameter of pores.

5.12.5 Titration equipment with galvanometer for photoelectric end point determination.

5.13 Additional apparatus required for conductimetric determination of free lime (see 18.2)

5.13.1 Measuring vessel (volume approximately 160 ml) with a thermoplastic casing and screw cap with 2 apertures NS 14 (see figure **2**).

5.13.2 Conductive electrode with ground cone NS 14.

5.13.3 Thermometer (50 °C to 100 °C): 0,1 °C graduated, with ground cone NS 14.

5.13.4 Conductance meter

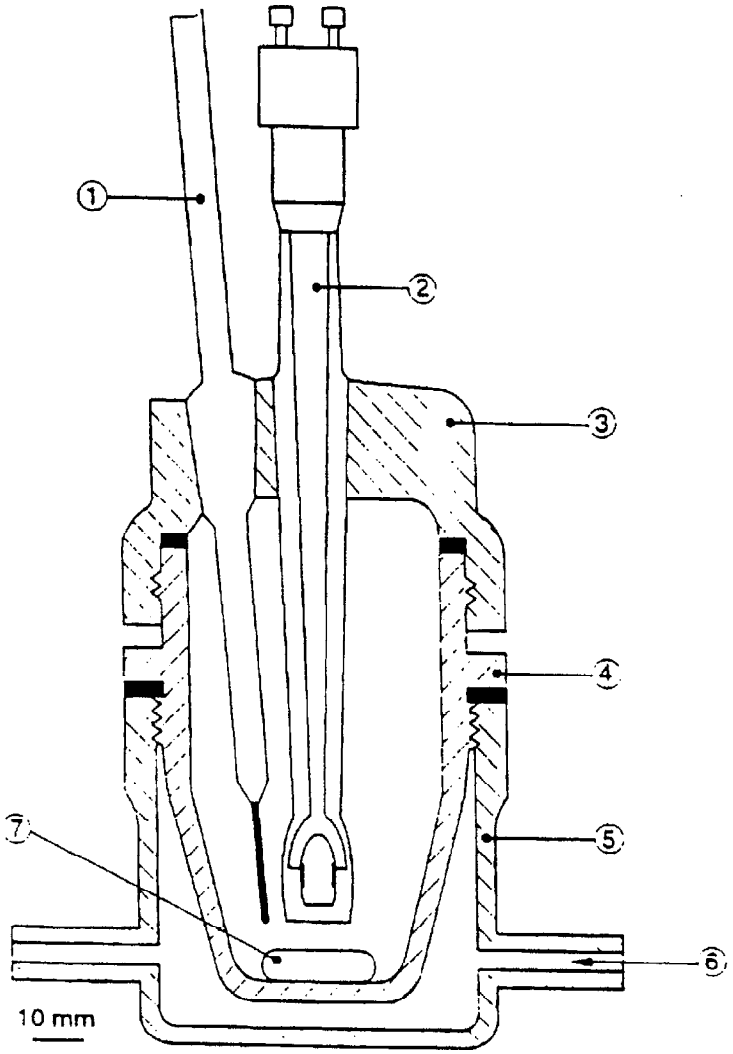
NOTE 1: The free lime content is obtained from the measured conductance using a calibration graph. This is established by dissolving known quantities of calcined CaO in ethanediol and measuring the conductance of their solutions. For this the conductance of at least five different solutions within the range from 0 mg to 10 mg CaO/100 ml ethanediol (**4.11.1**) should be determined, in each case with three individual measurements.

NOTE 2: The CaO used is produced by calcining $CaCO_3$ (**4.11.11**) at 1000 °C until it reaches a constant mass and then cooling in a desiccator which contains absorption materials for water and carbon dioxide, e.g. soda-lime.

NOTE 3: The conductance of the blank solution of ethanediol should always be established and deducted from the test solution.

NOTE 4: Figure **3** shows the calibration of an ethanediol solution containing calcium oxide at 80 °C and an electrode constant of 0,573 cm^{-1}; in this case, a measured conductance of 100 mS yields a value of 4,9 mass % for the content of free lime.

5.13.5 Water bath controllable to (80 ± 0,1) °C.



1 thermometer (**5.13.3**)
2 electrode (**5.13.2**)
3 polypropylene cap
4 vessel made of borosilicate glass
5 plastic casing
6 water inlet
7 magnetic stirrer (**5.12.3**)

Figure 2: Vertical section of a measuring vessel for conductometric determination of free lime (5.13 and 18.2)

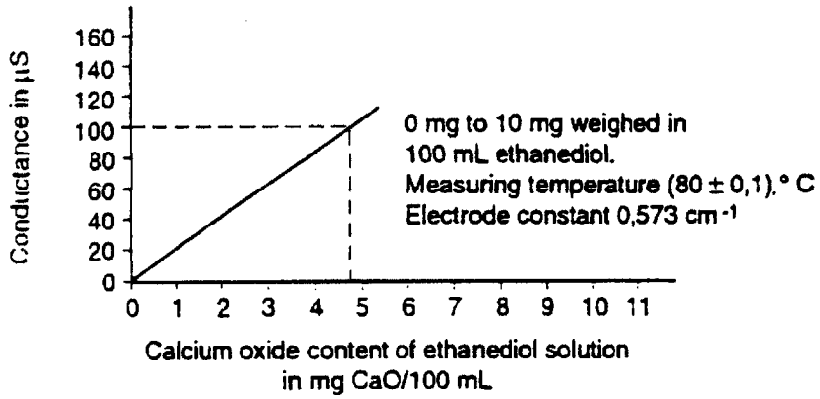


Figure 3: Example of a calibration graph

5.14 Additional apparatus required for acidimetric determination of free lime (see 18.3)

5.14.1 Erlenmeyer flasks, 200 ml, 250 ml or 300 ml capacity, fitted with water-cooled condensers by means of standard-taper inner joints.

5.14.2 Absorption tubes to fit to the upper part of the condensers and containing the sodium hydroxide (**4.13.8**) and the molecular sieve (**5.14.3**).

5.14.3 Molecular sieve 0,3 nm beads about 2 mm diameter.

5.14.4 Glass microfibre filters of 1,2 mm retention.

5.14.5 Equipment for vacuum filtration.

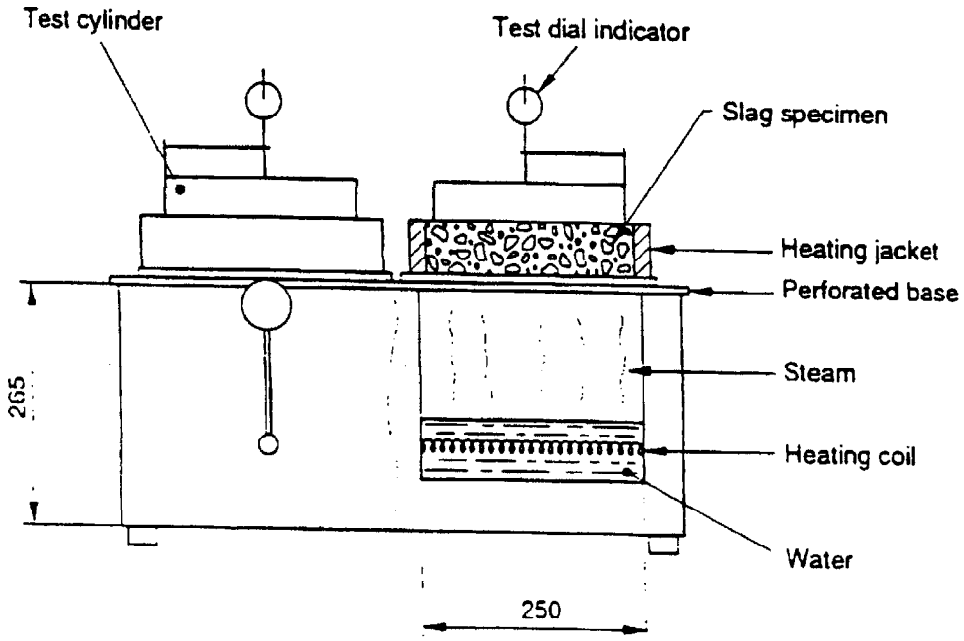
5.15 Additional apparatus required for the determination of dicalcium silicate disintegration of air cooled blast-furnace slag (see 19.1)

5.15.1 Ultra-violet lighting equipment, of wavelength 300 nm to 400 nm with a maximum wavelength intensity at 366 nm.

5.16 Additional apparatus required for determination of the volume expansion of steel slag (see 19.3)

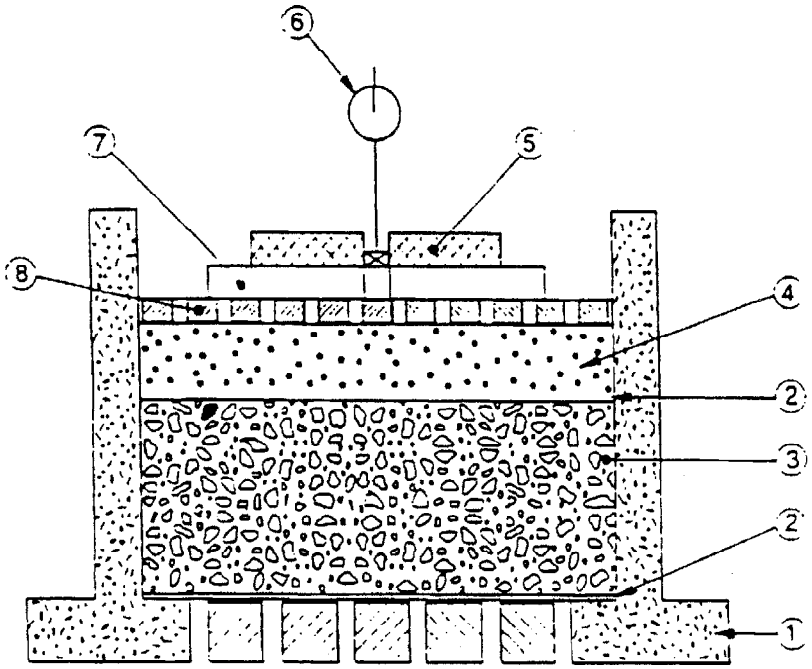
5.16.1 Steam unit with test cylinder and dial test indicator or displacement meter with a measuring range of (10 ± 0,01) mm as shown in figures **4** and **5**.

NOTE: The steam equipment consists of two chambers, in which water is heated up to boiling point over heating elements during the test. The maximum output of the heating elements is 2 kW. Above the heating chamber is the compressed slag specimen in a cylinder with a perforated base (cylinder diameter 210 mm, cylinder height 100 mm). The steam arising when heated up can therefore flow through the specimen evenly. In order to prevent condensation building up on the inside of the cylinder due to the heat loss, the cylinder is heated to (120 ± 10) °C by a circular heating jacket fitted to the outside wall (nominal output 250 Watts).



All dimensions in millimetres

Figure 4: Vertical section of a typical steam equipment



1 cylinder with perforated base, 0,01 holes per cm^2
e.g. 49 holes, 3 mm diameter distributed as follows:
central point: 1 hole
circle, 65 mm diameter: 8 holes
circle, 125 mm diameter: 16 holes
circle, 185 mm diameter: 24 holes
2 fabric mat
3 compacted slag specimen
4 glass beads, 5 mm diameter
5 loading weight
6 dial indicator
7 weighting cross
8 perforated plate, 0,3 holes per cm^2,
e.g. 3 mm diameter holes on concentric circles 6 mm distant,
at 6,5 mm to 7 mm intervals on each circle

Figure 5: Diagrammatic illustration of the testing device for determining expansion

5.16.2 Sieves, complying with EN 933-2, with mesh sizes 0,5 mm, 2,0 mm, 5,6 mm, 8,0 mm, 11,2 mm, 16,0 mm and 22,4 mm.

5.16.3 Glass beads, diameter 5 mm.

5.16.4 Medium grade filter paper, diameter 240 mm.

5.16.5 Vibrating table, with an approximate frequency of (48 ± 3) Hz and an amplitude of ± 1,5 mm, or a manual compacting apparatus such as a Proctor hammer or a hand-held power hammer which allows a final compaction of the test portion to a void content between 20 % volume and 25 % volume (see EN 196-1).

5.16.6 Sounding rod graduated to the nearest mm, with a minimum total scale of 200 mm.

5.16.7 Loading mass with an exterior diameter less than 210 mm (e.g. 180 mm) and having a central hole (e.g. 15 mm diameter), giving a way to the stem of the indicator and to the steam; the total mass of "loading mass plus weighting cross plus glass beads" to be 6 kg.

6 General requirements for testing

6.1 Number of tests

Unless otherwise stated the number of single determinations of the various test methods (see clauses **7** to **19.3**) is fixed at two (see also **6.3**).

6.2 Repeatability and reproducibility

The standard deviation of repeatability gives the closeness of agreement between successive results obtained with the same method on identical material tested under the same conditions (same operator, same apparatus, same laboratory and short time interval).

The standard deviation of reproducibility gives the closeness of agreement between individual results obtained with the same method on identical material but tested under different conditions (different operators, different apparatus, different laboratories and/or different times) (see prEN 932-6).

The standard deviations of repeatability and reproducibility are expressed in absolute percentage.

6.3 Expression of mass, volume, factors and results

Record the mass from an analytical balance (**5.2.6**) in grams to the nearest 0,1 mg and volume from the burettes (**5.2.13**) in millilitres to the nearest 0,05 ml.

Record the mass from the ordinary balance specified in **5.2.4** in grams to the nearest 1 g or from the balance specified in **5.2.5** to the nearest 0,01 g.

Express the factors of solutions (**4.7.5**, **4.7.6** and **4.13.7**), given by the mean of three determinations, to three decimal places.

Express the results of the tests, given by the mean of two determinations, as a percentage, to the nearest 0,01 %, unless otherwise stated.

If the difference between two determinations is more than twice the repeatability standard deviation, repeat the test and take the mean of the two closest values.

6.4 Drying of materials

Drying shall be carried out in a well ventilated oven (**5.2.1**), at a temperature of (110 ± 5) °C.

6.5 Determination of constant mass after drying

Constant mass is assumed after a test portion is dried for a period of 24 h.

6.6 Ignitions of precipitates

Carry out the ignitions as follows.

Place the filter paper and its contents into a crucible which has been previously ignited and tared. Dry it, then incinerate it slowly in an oxidizing atmosphere, without flaming while ensuring complete combustion.

Ignite for at least 1 h at the stated temperature. Allow the crucible and its contents to cool to the room temperature in a desiccator. Weigh the crucible and its contents.

6.7 Check for the absence of chloride ions (silver nitrate test)

After generally five to six washes of a precipitate, rinse the base of the filter stem with a few drops of water.

Wash the filter and its contents with several millilitres of water and collect this in a test tube. Add several drops of concentrated nitric acid (**4.1**) and of silver nitrate solution (**4.2.1**). Check the absence of turbidity or precipitate in the solution. If present, continue washing, carrying out periodic checks. The absence of turbidity in the silver nitrate test confirms that the washings are free from chloride ions.

7 Determination of water-soluble chloride salts using the Volhard method (Reference method)

7.1 Principle

This test is suitable for aggregates where the chloride content derives directly from contact with, or immersion in, saline water, e.g. typical sea-dredged aggregates. With some aggregates, e.g. from some desert areas, testing a nitric acid extract of the finely ground aggregate can show significantly higher levels of chloride than the water extraction method specified in this clause.

An aggregate test portion is extracted with water to remove chloride ions. The method of analysis of the extract is based on that of Volhard titration where an excess of silver nitrate solution is added to the chloride solution and the unreacted portion is back-titrated with a standardized solution of thiocyanate, using ammonium iron (III) sulfate solution as an indicator.

The chlorides are expressed in terms of, and reported as, the chloride ion content as a percentage by mass of the aggregate.

7.2 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.

7.3 Preparation of test portion

Reduce the laboratory sample by the procedures specified in prEN 932-2 to an amount not less than the mass given in table **1** appropriate to the nominal size of aggregate.

Dry the sub-sample at a temperature of (110 ± 5) °C to constant mass (**6.5**).

Sieve the sub-sample through a 16 mm sieve (see **5.3.1**) and crush any oversize samples to pass the sieve avoiding excessive grinding. Combine, mix thoroughly and using the procedures specified in prEN 932-2 produce two test portions each of about (2 ± 0,3) kg mass for coarse aggregates or two test portions each of about (500 ± 75) g mass for fine aggregates.

In the case of lightweight aggregates, the two test portions will be about 1 l.

Table 1: Minimum mass of preliminary sub-sample

Nominal maximum particle size of aggregate mm	Minimum mass of sub-sample kg
63	50
45	35
22,4 or less	15
	5

7.4 Preparation of extracts

For coarse aggregates and lightweight aggregates use the two wide-mouthed glass, plastic or metal bottles of 5 l capacity and for fine aggregates use the two bottles of 2 l capacity (**5.3.2**). Weigh each bottle and record its mass to the nearest 1 g.

Transfer the test portions obtained, as specified in **7.3**, to the bottles, weigh bottles and contents and record their mass to the nearest 1 g. Calculate the mass of aggregate in each bottle by difference.

Add to each bottle a mass of water equal to the mass of the test portion. For lightweight aggregates add 1 l water.

Agitate the bottles continuously for 60 min by means of the shaker or the roller (**5.3.3**).

Then filter the extracts through dry, medium grade filter papers (**5.3.4**) until at least 100 ml of clear or slightly opalescent filtrates have been collected in clean dry beakers (**5.2.9**).

7.5 Procedure for the determination of the chloride content of the extracts

Take 100 ml of the filtered extract (**7.4**) by means of the 100 ml pipette (**5.2.10**) and transfer to the 250 ml capacity flask (**5.3.5**). Add 5 ml of nitric acid (**4.2.3**) to the flask followed by silver nitrate solution (**4.2.1**) from a burette (**5.2.13**) until all the chloride has been precipitated and then add excess.

> NOTE: When aggregates containing sulfides (e.g. slags) are being analyzed, allow the solution to digest for 3 min or 5 min at a temperature just below boiling. A white precipitate of sulfur may form, but it is not necessary to filter this off. Cool and add the silver nitrate solution.

Sufficient silver nitrate is required to ensure a titre of at least 3 ml of thiocyanate solution.

Note the total volume V_5 of silver nitrate solution added.

Add 2 ml of 3,5,5-trimethylhexan-1-ol (**4.2.4**), stopper, and shake the flask vigorously to coagulate the precipitate.

Carefully loosen the stopper, avoiding loss of solution and rinse the stopper with water, collecting the washings in the solution.

Add 5 ml of the ammonium iron (III) sulfate indicator solution (**4.2.5**) followed by the standardized thiocyanate solution (**4.2.2**) from a burette until the first permanent colour change occurs, that is from white opalescence to pale brown and the solution has the same depth of colour as was used for the standardization specified in **4.2.2**.

Note the volume V_6 of the thiocyanate solution added.

Repeat the procedure with the extract from the second test specimen.

The number of determinations on each extract is fixed at one. The result of the test is given as the mean of the determinations on the two extracts.

7.6 Calculation and expression of results

Calculate the chloride content C of aggregate from the following equation:

$$C = 0{,}003546W\{V_5 - (10 \times c_T \times V_6)\} \quad \text{(in \%)}$$

where:

V_5 is the volume of silver nitrate solution (in millilitres);
V_6 is the volume of standardized thiocyanate solution added (in millilitres);
c_T is the concentration of the standardized thiocyanate solution (in moles per litre);
W is the water/aggregate ratio (in grams/gram) - for lightweight aggregates, W is 1000 g/mass of the aggregate in grams.

NOTE: A statement on the precision of the determination of water-soluble chlorides is given in annex **A**.

8 Determination of water-soluble chloride salts by potentiometry (Alternative method)

8.1 Principle

The aggregate test portions are extracted in the same way as in 7.4. The chloride ions are precipitated from the extracts by means of a standard silver nitrate solution.

The titration is executed by potentiometry, using a suitable electrode as indicator.

NOTE: Chloride ion selective electrodes and the use of Gran's plot are also permitted (see annex **B**).

8.2 Sampling, preparation of test portions and extracts

Follow the procedures specified in **7.2**, **7.3** and **7.4**.

8.3 Procedure for the determination of the chloride content of the extracts

Take 50 ml of the filtered extract (**7.4**) by means of the 50 ml pipette (**5.2.10**) and transfer to a 250 ml beaker. Acidify with nitric acid (HNO_3) (**4.2.3**) to a pH value of 2 to 3. Add by pipette 5 ml of sodium chloride solution (**4.3.2**).

NOTE: When aggregates containing sulfides (e.g. slags) are being analyzed, allow the solution to digest for 3 min to 5 min at a temperature just below boiling. A white precipitate of sulfur may form, but it is not necessary to filter this off. Cool and proceed with the titration.

By means of the potentiometric device (**5.4.1**), titrate with the silver nitrate solution (**4.3.1**). The chloride content of the solution is indicated by the consumption of silver nitrate solution relating to the point of inflection of the potential curve, the quantity of sodium chloride (**4.3.2**) added to improve end point recognition having been deducted.

Repeat the procedure with the extract from the second test portion.

A blank test shall be carried out to confirm the amount of sodium chloride added.

8.4 Calculation and expression of results

Calculate the chloride content C of aggregate from the following equation:

$$C = 0{,}000709 V_7 \times W \text{ (in \%)}$$

where:

V_7 is the consumption of silver nitrate solution, in millilitres, subtracting 10 ml for the added chloride solution;
W is the water:aggregate ratio (in grams/gram) - for lightweight aggregates, W is 1000 g/mass of the aggregate in grams.

NOTE: A statement on the precision of the determination of water-soluble chlorides by potentiometry is given in annex **A**.

9 Determination of water-soluble chloride salts using the Mohr method (Alternative method)

9.1 General

This test method gives a more rapid method of extraction than the one specified in clause 7. It is strongly recommended that this procedure is used as a preliminary check before resorting to the test specified in clause 7 which may be needed for compliance with a specification. This procedure shall only be used for factory production control.

The chloride ion concentration of the aqueous extract of natural aggregate may be determined using instrumental techniques based on conductivity measurement.

9.2 Principle

An aggregate test portion is rapidly extracted with water at room temperature to remove chloride ions. The extract is then analysed by the method based on Mohr, whereby the chloride is titrated with silver nitrate, potassium chromate being used as an indicator. The chloride ion concentration may also be determined using instrumental methods based on conductivity.

9.3 Sampling

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

9.4 Preparation of test portion

Reduce the laboratory sample to a test portion of 250 g of aggregate (1 l in the case of lightweight aggregate) using the procedures specified in prEN 932-2.

9.5 Preparation of extracts

For coarse and fine aggregates use the wide-mouthed plastic bottles of 1 l capacity (**5.5.1**). For lightweight aggregate use the bottle of 5 l capacity (**5.3.2**). Weigh each bottle and record its mass to the nearest 1 g.

Transfer the test portion into the bottles, weigh bottles and contents and record their mass to the nearest 1 g. Calculate the mass of aggregate in each bottle by difference.

Add to each bottle a mass of water equal to the mass of aggregate. For lightweight aggregate add 1 l water.

Fasten the water-tight bottle closures and mix the contents by shaking at least 20 times. Allow to settle until the supernatant water is more or less clear.

9.6 Procedure for the determination of the chloride content of the extracts

NOTE: If conductivity measurement is being used, decant approximatively 100 ml into a 250 ml beaker and measure the concentration.

Take 25 ml of the supernatant water (**9.5**) by means of the 25 ml pipette (**5.2.10**) and transfer to the 100 ml capacity flask (**5.3.5**).

Add 4 ml to 6 ml of the potassium chromate solution (**4.4.1**) and mix. Titrate by means of the 0,01 mol/l silver nitrate solution (**4.3.1**) until light red. Note the volume V_8 of the silver nitrate solution used.

9.7 Calculation and expression of results

Calculate the chloride content C of the aggregate from the following equation:

$$C = 0{,}01 \times 0{,}03545 \times V_8 \times W \times 4 \quad (\text{in } \%)$$

where:

V_8 is the used volume of 0,01 mol/l silver nitrate solution;
W is the water/aggregate ratio (in grams/gram) - for lightweight aggregates, W is 1000 g/mass of the aggregate in grams.

10 Determination of water-soluble sulfates

10.1 Principle

An aggregate test portion is extracted with water to remove water-soluble sulfate ions. The water-soluble sulfate content is determined by precipitation at pH between 1 and 1,5 by a solution of barium chloride, at the boiling point.

The determination is then completed gravimetrically and the sulfate ion content is expressed as a percentage by mass of the aggregate.

This method is applicable when aggregates containing sulfides, e.g. slags, are being analyzed.

10.2 Sampling

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

10.3 Preparation of test portion

Reduce the laboratory sample by the procedures specified in prEN 932-2 to an amount not less than the mass given in table 1 (**7.3**) appropriate to the nominal size of aggregate.

Dry the sub-sample at a temperature of (110 ± 5) °C to constant mass (**6.5**).

Sieve the sub-sample through a 16 mm sieve (see **5.3.1**) and crush any oversize particles to pass the sieve avoiding excessive grinding. Combine, mix thoroughly and using the procedures specified in prEN 932-2 produce two test specimens each of about (2 ± 0,3) kg mass for coarse aggregates or two test specimens each of about (500 ± 75) g mass for fine aggregates.

In the case of lightweight aggregates, the two test specimens will be about 1 l.

10.4 Preparation of extracts

For coarse or lightweight aggregates use the two plastics or metal bottles of 5 l capacity and for fine aggregate use the two bottles of 2 l capacity (**5.3.2**). Weigh each bottle and record its mass to the nearest 1 g.

Transfer the test specimens obtained as specified in **10.3** to the bottles, weigh the bottle and contents and record their mass to the nearest 1 g. Calculate the mass of aggregate in each bottle by difference.

Add to each bottle a mass of water equal to twice the mass of the test specimen. For lightweight aggregate add 1 l water. Fasten the water-tight bottle closures and mix the contents by shaking or rolling (**5.3.3**) continuously for a minimum of 24 h.

> NOTE: If mechanical agitation equipment is not used and the aggregate and water are merely allowed to remain in contact with occasional shaking there is a possibility (particularly if the source of sulfate is, for example, large crystals of gypsum) that the sulfate which could theoretically dissolve will not all be extracted in 24 h.

Filter the extracts through dry, medium-grade filter papers (**5.3.4**) until at least 100 ml of clear filtrates have been collected in clean dry beakers (**5.2.9**).

10.5 Procedure for the determination of the sulfate content of the extracts

With a pipette (**5.2.10**), transfer 50 ml of filtered extract to a 500 ml beaker, dilute to 300 ml with water, add 10 ml of hydrochloric acid solution (**4.5.1**).

Bring to the boil and boil for 5 min.

> NOTE: If the aggregate contains sulfides, e.g. slags, after boiling for 5 min, stand the solution in a warm place for 30 min. If a white precipitate forms filter through a medium grade filter paper and wash thoroughly with hot distilled water, continue as usual discarding the residue.

While stirring vigorously, the solution maintained at boiling point, add drop by drop 5 ml of the barium chloride solution (**4.5.2**) heated to just below boiling. Continue the boiling for 15 min so that the precipitate is properly formed.

Stand just below boiling for 30 min then leave in a warm place overnight.

Transfer the precipitate of barium sulfate with extreme care to a previously ignited and weighed sintered silica filter crucible (**5.6.1**) using suction. Alternatively transfer the precipitate with extreme care to a fine filter paper in a glass funnel and filter. In either case wash the precipitate several times with hot water until the washings are free from chloride (**6.7**).

If a sintered silica filter crucible is used remove it from the filter flask and dry at (110 ± 5) °C for approximately 30 min and gradually raise the temperature to (925 ± 25) °C in an electric muffle furnace (**5.2.2**) until no further loss in mass occurs; 15 min at this temperature should suffice.

Cool the crucible in a desiccator (**5.2.16**) and weigh to the nearest 0,1 mg, and calculate the mass of the precipitate m_3 from the increase in mass of the crucible.

If the precipitate is filtered through a filter paper, transfer the filter paper and precipitate to a previously ignited and weighed crucible (**5.6.2**). Place the crucible and contents in the electric muffle furnace (**5.2.2**) following the procedure specified in **6.6**.

Calculate the mass of the precipitate m_3 from the increase in mass (to the nearest 0,1 mg) of the crucible.

10.6 Calculation and expression of results

Calculate the soluble sulfate content of the aggregate, expressed as SO_3, from the following equation:

$$\text{Soluble } SO_3 = 2 \times W \times 0{,}343 \times m_3 \quad (\text{in } \%)$$

where:

m_3 is the mass of the precipitate of barium sulfate in grams;
W is the water: aggregate ratio (in grams/gram) - for lightweight aggregates,
W is 1000 g/mass of the aggregate in grams.

11 Determination of total sulfur content

11.1 Principle

An aggregate test portion is treated with bromine and nitric acid to convert any sulfur compounds present to sulfates; the sulfates are precipitated and weighed in the form of $BaSO_4$. The sulfur content is expressed as a percentage by mass of the aggregate.

11.2 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.

11.3 Preparation of test portion

Reduce the laboratory sample by the procedures specified in prEN 932-2 to an amount not less than the mass specified in table 1 appropriate to the nominal size of the aggregate.

If necessary, dry the sample at a temperature not exceeding (110 ± 5) °C to avoid the oxidation of sulfides.

Stepwise crush and reduce the sub-sample to a mass of approximately 20 g; grind this sample until it passes the 125 µm sieve. Take approximately 1 g of this material as the test portion.

11.4 Procedure

Weigh the test portion to the nearest 0,1 mg (m_4) and transfer into a wide-mouthed Erlenmeyer flask (**5.12.1**) in the neck of which rests a short-stemmed funnel. Add to the flask in the fume cupboard (**5.2.17**) 3 ml of water and 1 ml of bromine (**4.6.1**) and gently agitate the mixture for 1 min to prevent undue formation of lumps, then slowly add through the funnel 15 ml of sulfur free concentrated nitric acid (**4.1**). Allow the mixture to stand on a steam bath for 1 h and break up the gel at intervals with a flattened glass rod, leaving the rod in the flask for this purpose. Add 30 ml of water and gently boil the mixture on a hot plate (**5.2.7**) until evolution of the denser brown fumes ceases. Add 5 ml of concentrated hydrochloric acid (**4.1**) and 10 ml of water and reduce by boiling the mixture to a small volume. Repeat the addition and reduce by boiling the mixture to a small volume. Transfer the contents of the flask to a 250 ml beaker (**5.2.9**)and wash the flask until the total volume in the beaker is approximately 100 ml.

Add a little filter paper pulp, bring the contents of the beaker nearly to the boil; make the solution alkaline by adding ammonia, check the alkalinity using methyl red indicator (**4.6.2**) or by means of a pH-meter (**5.2.8**). Simmer for 30 s, filter under gentle suction (using a filter paper of medium porosity) and wash once with a little hot, distilled water, reserving the filtrates. Transfer the filter paper to the beaker and redissolve it in 5 ml concentrated hydrochloric acid to which has been added 70 ml hot water.

Proceeding as above, boil, precipitate, filter and wash, rejecting the precipitate. Acidify the combined filtrates and washings (which should be about 220 ml in all) with 1 ml concentrated hydrochloric acid, bring to the boil and boil for 5 min. While stirring vigorously the solution maintained at boiling point, 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 **10.5**.

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

11.5 Calculation and expression of results

Calculate the total sulfur content of the aggregate, expressed as S, from the following equation:

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

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**.

12 Determination of acid soluble sulfates

12.1 Principle

The sulfates, extracted from a test portion of the aggregate by dilute hydrochloric acid, are determined by gravimetry. The sulfate ion content is 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.

Ensure that the laboratory sample is representative of the moisture content as well as the solids.

12.3 Preparation of test portion

Reduce the laboratory sample by the procedures specified in prEN 932-2 to an amount not less than the mass specified in table 1 appropriate to the nominal size of the aggregate. Stepwise crush and reduce the sub-sample. Then proceed to grinding and further reduction until a mass of approximately 20 g passing the 0,125 mm test sieve is obtained. Take approximately 2 g of this material as the test portion.

If drying is necessary during the procedure, the temperature shall not exceed (110 ± 5) °C to avoid the oxidation of sulfides.

12.4 Procedure

Weigh the test portion to the nearest 0,1 mg (m_6); place in a 250 ml beaker, add 90 ml of cold water. While stirring the mixture vigorously, add 10 ml of concentrated hydrochloric acid. Heat the solution gently and break up the solids with the flattened end of a glass stirring rod. Allow the solution to digest for 15 min at a temperature just below boiling.

NOTE: Aggregates containing significant amounts of carbonate will froth on addition of the acid. In these cases, add the acid slowly while continuously stirring. Aggregates containing sulfide will release H_2S on acidification and this will be noticeable by its smell. In these cases there is a danger that this procedure will overestimate the sulfate content because of sulfide oxidation. To avoid any oxidation, place 90 ml water and 10 ml of concentrated hydrochloric acid in a 250 ml beaker and heat to boiling point. Remove from the source of heat and, while stirring, sprinkle the test portion on to the acid solution.

Filter the residue through a medium filter paper into a 400 ml beaker. Wash thoroughly with hot water. Check the washings for the absence of chloride ions by the silver nitrate test (**6.7**).

Adjust the volume to about 250 ml; if necessary, 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 min. Check that the solution is clear; if not, start the test again using a new test portion. While stirring vigorously the solution maintained at boiling point, 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 **10.5**.

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, from the following equation:

$$\text{Sulfate content} = m_7/m_6 \times 34{,}30 \quad (\text{in } \%)$$

where:

m_7 is the mass of precipitate in grams;
m_6 is the mass of the test portion in grams.

NOTE: A statement on the precision of the determination of the acid soluble sulfate content is given in annex **A**.

13 Determination of acid soluble sulfides

13.1 Principle

The test portion is decomposed by hydrochloric acid under reducing conditions. The sulfides are converted into hydrogen sulfide, which is carried over by a gaseous stream into an ammoniacal solution of zinc sulfate. The precipitated zinc sulfide is determined by iodometry.

13.2 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.

13.3 Preparation of test portion

Reduce the laboratory sample by the procedures specified in prEN 932-2 to an amount not less than the mass specified in table **1** appropriate to the nominal size of the aggregate. Stepwise crush and reduce the sub-sample. Then proceed to grinding and further reduction until a mass of approximately 20 g passing the 0,125 mm test sieve is obtained. Take approximately 1 g of this material as the test portion.

If drying is necessary during the procedure, the temperature shall not exceed (110 ± 5) °C to avoid the oxidation of sulfides.

13.4 Procedure

Use the apparatus depicted in **5.7.1** and figure **1**. Weigh the test portion to the nearest 0,1 mg (m_8) and transfer into a 250 ml stoppered round bottom flask with a ground glass joint.

> NOTE 1 : If the sulfide content is low (<0,1 %), reagent solutions ten times more dilute should be used (**4.7.5** and **4.7.6**).

Add about 2,5 g of tin (II) chloride (**4.7.3**) and 0,1 g of chromium (**4.7.4**).

> NOTE 2 : Chromium contributes to the dissolution of any pyrites (FeS_2) that happens to be present in the aggregate.

Disperse the mixture in 50 ml of water. Fix to the flask the ground neck supporting the separating funnel and connect this neck to the inlet of the condenser; connect the outlet of the condenser to the glass tube which dips into the beaker containing 15 ml of ammoniacal zinc sulfate solution (**4.7.2**) and 285 ml of water. Connect the gas supply (nitrogen or argon) and adjust the flow to about 10 ml per min. Stop the flow of gas. Release 50 ml of hydrochloric acid (1 + 1) from the separating funnel taking care that a small amount of acid remains in the separating funnel to prevent leakage. Reconnect the gas supply, heat the contents of the flask to boiling and boil for 10 min. Disconnect the outlet tube which will serve as a stirrer during the titration.

> NOTE 3 : Some aggregates of higher sulfide content may require more than 10 min reaction time for complete transformation of all sulfides to precipitated zinc sulfide. Check that the extraction is complete by bubbling the outlet into fresh ammoniacal zinc sulphate solution. If no precipitate appears, the extraction is complete.

Cool the receiver to 20 °C and add by pipette 10 ml of the 0,0166 mol/l potassium iodate solution (**4.7.5**) and 25 ml concentrated hydrochloric acid. Titrate with sodium thiosulfate solution (**4.7.6**) until pale yellow. Then add 2 ml of starch solution (**4.7.7**) and titrate until the colour changes from blue to colourless.

13.5 Calculation and expression of results

Calculate the sulfide content of the aggregate, expressed in S, from the following equation:

$$S = \frac{\{(V_9 \times F) - (V_{10} \times f)\} \times 1{,}603 \times 100}{1000 \times m_8} = 0{,}1603 \frac{(V_9 \times F) - (V_{10} \times f)}{m_8} \quad \text{(in \%)}$$

where:

V_9 is the volume of the potassium iodate solution in millilitres;
F is the factor of the potassium iodate solution as specified in **4.7.5**;
V_{10} is the volume of sodium thiosulfate solution used for the titration in millilitres;
f is the factor of the sodium thiosulfate solution as specified in **4.7.6**;
m_8 is the mass of the test portion in grams.

14 Determination of components affecting the surface finish of concrete

14.1 Examination for the presence of reactive iron sulfide particles

14.1.1 General

This clause specifies methods for detection of iron sulfide particles which, when present at or near the surface of concrete, may cause the formation of brown stains. These stains may be impossible to remove except by cutting out.

14.1.2 Sampling

The stockpile shall be inspected and approximatively 50 particles, representative of those suspected to contain iron sulfide, shall be taken for testing.

14.1.3 Procedure

Determine the reactivity of the particles by placing them in a saturated limewater solution.

NOTE: A blue-green gelatinous precipitate of ferrous sulfate should form within 5 min. This precipitate changes rapidly to brown ferric hydroxide on exposure to air and light. This reaction should be complete within 30 min and indicates the presence of rapidly reacting iron sulfide.

If no brown gelatinous precipitate is formed when the particles are placed in saturated limewater they may be slowly reacting particles. If the presence of slowly reacting particles is suspected the following procedure shall be carried out.

Examine the particles visually to assess as well as possible their propensity to cause staining in mortar or concrete.

NOTE: Where a particular quarry has a history of occasionally producing aggregate containing iron sulfide, examination with a low power microscope by a technologist with experience of the quarry may be sufficient to determine whether or not the material is deleterious.

In other cases, embed the recovered particles in Portland cement paste, store for 28 days in a moist condition and then examine the cement pat for staining.

14.2 Determination of lightweight contaminators

14.2.1 General

This test is a method for estimating the mass percentage of lightweight particles in fine aggregate. The method estimates substances such as lignite and coal which may cause staining or pop-outs on concrete or mortar surfaces. If necessary, the method can be adapted for use on coarse aggregates by examining larger test portions (see table 1).

14.2.2 Principle

The inorganic minerals which comprise commercially available fine aggregates for concrete and mortar almost without exception have a relative particle density greater than 2,0. By immersing a suitable test portion of the fine aggregate in a liquid of relative density just below 2,0, lower density particles can be caused to float on the liquid facilitating their removal for examination and quantification.

NOTE: This flotation process is inapplicable to lightweight aggregates and the determination of lightweight contaminators in lightweight aggregates should be carried out by hand-picking.

14.2.3 Procedure

The minimum quantity of fine aggregate in the laboratory sample shall be 5 kg; this shall be reduced to produce a test portion of mass (350 ± 50) g.

Spread the test portion over the tray (**5.2.1**) and transfer it to the oven at (110 ± 5) °C (**6.4** and **6.5**). Record the mass of the dried sand (m_9) to the nearest 0,1 g. Separate the aggregate on the 300 µm sieve (**5.8.1**), discarding the finer fraction.

Pour about 1 l of the solution of zinc chloride (**4.8.1**) or sodium polytungstate (**4.8.2**) into a 2 l beaker and then pour the aggregate into the solution. Gently agitate the bed of aggregate with a glass rod to assist lightweight particles to leave the bed and rise to the surface of the solution. Gently agitate the floating particles with the glass rod to dislodge air bubbles from any floating aggregate particles, and allow these particles to sink to the aggregate bed.

Decant the supernatant solution into a second 2 l beaker passing the liquid through the 250 µm sieve (**5.8.1**), while allowing the floating particles to pass into, and be collected by, the sieve. Ensure that no aggregate passes into the sieve. Return the solution to the first beaker and agitate the bed of aggregate again. If further particles float on the solution, decant through the sieve again and repeat the process until all floating particles have been collected in the sieve.

Wash the sieve and the particles on it with water until the zinc chloride or sodium polytungstate has been removed. Dry the sieve and contents for (20 ± 4) h at (40 ± 5) °C and then tip the contents into an evaporating basin (**5.8.2**) and complete the drying at (110 ± 5) °C for (4 ± 0,25) h.

Cool the basin and then weigh the lightweight particles (m_{10}), to the nearest 0,1 g.

14.2.4 Calculation and expression of results

Calculate the percentage of lightweight particles in the aggregate, expressed as m_{LPC}, from the following equation:

$$m_{LPC} = m_{10}/m_9 \times 100 \quad (\text{in } \%)$$

where:

m_9 is the mass of the oven dried test portion in grams;
m_{10} is the mass of oven dried lightweight particles separated from the test portion in grams.

The result shall be recorded to the nearest 0,1 %.

15 Determination of organic components affecting the setting and the hardening of cement

15.1 Determination of humus content

15.1.1 Principle

Humus is an organic substance which forms in the ground by the decomposition of animal and plant residues.

The content of humus is estimated from the colour formed when a test portion is shaken in a sodium hydroxide solution.

NOTE: The method is based on the fact that humus develops a dark colour by reaction with NaOH. The intensity of the colour depends on the humus content. If the solution is not or only slightly coloured, the aggregate does not contain considerable amounts of humus. A strong colour reaction will usually derive from a high content of humus, but may be due to other things. Therefore, in this case the method does not allow any definitive conclusion.

15.1.2 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.

15.1.3 Preparation of test portion

Dry the sub-sample (**7.3**) spread over the trays in the oven (**5.2.1**) at (55 ± 5) °C instead of (110 ± 5) °C . Sieve the sample on the 4 mm sieve (**5.9.1**) and keep the fraction retained on the sieve. Crush the fraction retained on the sieve to less than 4 mm, and recombine with the material already passing 4 mm.

15.1.4 Procedure

Pour a 3 % NaOH solution (**4.9.1**) into the glass bottle (**5.9.2**) to a height of 80 mm. Then pour the test portion until the height of aggregate and solution is 120 mm. Shake the bottle to allow air bubbles to escape. Stopper the bottle and shake vigorously for 1 min and leave to stand. After 24 h compare the colour of the solution to the standard colour solution (**4.9.2**), contained in a similar bottle.

15.1.5 Expression of results

The test result shall state whether the colour of the solution is lighter or darker than the standard colour (**4.9.2**).

15.2 Determination of fulvo acid content

15.2.1 General

This clause specifies a method of determination of fulvo acid content that may be present in fine aggregates, especially in sandy soils to be stabilized with cement.

15.2.2 Principle

Fulvo acids are components of the humic acids which have a retarding effect on the hydration of cements. Fulvo acids dissolve in hydrochloric acid producing a yellow colour. The intensity of the colour increases as the concentration of fulvo acids increases. Compounds of Fe(III) produce a brown colour in hydrochloric acid. This colour is eliminated by converting Fe(III) compounds to colourless Fe(II) compounds using stannous chloride solution.

15.2.3 Sampling

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

Ensure that the labotatory sample is representative of the moisture content as well as the solids.

15.2.4 Preparation of test portion

Reduce the laboratory sample to a test portion of (100 ± 0,5) g using the procedures specified in prEN 932-2.

The moisture content of the test portion shall not be greater than 10% by mass of the sample. Test portions with moisture contents greater than 10% shall be dried at a temperature of (40 ± 5) °C until the moisture content is reduced to 10 % or less.

15.2.5 Procedure

The test is carried out at a temperature of (20 ± 2) °C. Weigh the test portion into a 250 ml or 300 ml Erlenmeyer flask (**5.2.9**). Add 100 ml of hydrochloric acid (**4.10.1**). Allow the flask and its contents to stand for 4 h, shaking it occasionally. From the flask, filter 75 ml of the solution into a 250 ml graduated cylinder (**5.2.11**). Using a 10 ml graduated cylinder (**5.2.11**) add 10 ml of clear stannous chloride solution (**4.10.2**). Let the 250 ml graduated cylinder and its contents stand for a further 1 h then fill to the 100 ml graduation mark with hydrochloric acid (**4.10.1**). Mix the contents of the cylinder using a stirring rod (**5.10.1**).

If after adding the stannous chloride the solution becomes turbid, sulfides are present and the test shall be repeated by boiling for 5 min on a hot plate (**5.10.4**) before adding the stannous chloride.

15.2.6 Expression of results

Determine the amount of fulvo acid by choosing a standard colour plate (**5.10.5**) that has the same colour as the solution. Table 2 gives the levels of acceptability of fulvo acid content by comparison with the standard colour plate.

Table 2: Levels of acceptability of fulvo acid content

Colour plate (5.10.5)	Acceptability for concrete sand		Acceptability for soil stabilisation
	Strength at 3 days	Strength at 28 days	
A	no influence	no influence	good
B	no influence	no influence	good
C	moderate decrease	no influence	good to moderate
D	moderate decrease	no influence	moderate
E	strong decrease	no influence	moderate to bad
F	strong decrease	moderate decrease	bad
G	strong decrease	strong decrease	bad

15.3 Determination of organic contaminators by mortar method

15.3.1 Principle

The mortar method is a performance test intended to demonstrate and quantify any effects which organic contaminators in aggregate may have on the stiffening and hardening of mortar. The principle of the method is to prepare two nominally identical mortars and test them for stiffening rate and compressive strength. One mortar contains the test aggregate as received while the other mix is prepared from a duplicate test portion which has been heated to destroy organic matter. The heated aggregate acts, effectively, as a control alongside which the original aggregate is compared. The stiffening test assesses acceleration or retardation of the setting of the mortar, while the 28 day strength indicates any longer term effect.

15.3.2 Sampling

The laboratory sample shall be taken in accordance with the procedures specified in EN 932-1. The minimum quantity of aggregate in the laboratory sample shall be 15 kg, more than required in table 1.

15.3.3 Preparation of test portions

Dry the laboratory sample by spreading it over trays and allowing it to dry naturally in laboratory air at ambient temperature. Using the procedures specified in prEN 932-2 further reduce the dried laboratory samples to produce test portions each of (1900 ± 100) g.

When reducing the dried laboratory sample to produce test portions for the mortar method, should a splitting operation produce two test portions each of mass less than 1,8 kg, then one test portion shall be split once, or twice, or if necessary three times to yield a sub-sample which when added to the first produces a combined mass of no more than 2 kg.

NOTE: For lightweight aggregate each test portion amounts to 1 l of aggregate.

15.3.4 Treatment of aggregate

Retain two of the four test portions obtained as specified in **15.3.3** in their original state and heat the other two using the following procedure:

(a) Pour a test portion into a weighed porcelain or silica basin (**5.11.2**), weigh and place in the muffle furnace (**5.11.7**) at ambient temperature.

NOTE: Should only a small furnace be available, each test portion may be divided into two or more parts and the parts weighed, heated and reweighed separately and recombined when cool.

(b) Raise the temperature of the furnace to (480 ± 25) °C in (4 ± 0,25) h.

(c) Maintain the temperature of (480 ± 25) °C for (4 ± 0,25) h and then allow the furnace to cool overnight. Weigh the basin and aggregate at ambient temperature and record the loss of mass.

Treat a second test portion in the same way.

15.3.5 Constituents

The cement shall be CEM I cement complying with the requirements of ENV 197-1. It shall be stored in an airtight container.

15.3.6 Mix quantities

15.3.6.1 General requirements and trial mixes

Each mortar mix shall contain either a test portion of the unheated aggregate, or a test portion of the aggregate previously heated as in **15.3.4**. Each mortar mix shall also contain CEM I cement of mass one quarter that of the aggregate mass in the mix. The cement shall be weighed to ± 1 g.

Ensure that the water content of the mortars containing the unheated aggregate shall be such as to produce a standard consistence, defined as a mean plunger (**5.11.3**) penetration of (23 ± 0,5) mm, when determined by the method given in EN 1015-4.

For lightweight aggregate use 300 g of cement for each mortar mix and 30 g for each preliminary test.

To establish the required water contents, prepare a series of trial mixes, using the unheated aggregate, varying the water content successively and measuring the consistence of each until the correct value of consistence is attained. Note the mass of water contained in this last mix and calculate the water-cement ratio of this mix. Reject the trial mixes.

The unheated test aggregate needs to possess the same moisture condition as the control aggregate at the same time of weighing before heating. Therefore, carry out the trial mixes and prepare mortar test specimens with the test aggregate on the same day as heating is started on the control aggregate.

NOTE: Control mixes will thus be prepared one day after the test mixes, and laboratory conditions should be as similar as practicable on the two mixing days.

15.3.6.2 Test mixes

Having calculated the mass of cement appropriate to each test portion of unheated aggregate, then from the water-cement ratio obtained in **15.3.6.1** calculate the water required for each mix and weigh to ± 0,5 g.

15.3.6.3 Control mixes

Ensure that the water-cement ratio of the heated aggregate control mortars is the same as that of unheated aggregate test mortars by first calculating the required mass of water for each mix as in **15.3.6.2.** Then add to each calculated mass, the mass lost by the corresponding portion of aggregate during the heating detailed in **15.3.4(c)**. Weigh this total of water to ± 0,5 g for each mix.

15.3.7 Mixing procedure

Four mixes are required; two prepared from the aggregate as received, but air dried, and two from the heated aggregate. Bring all the materials to a temperature of (20 ± 2) °C before starting the mixing of the mortar. Carry out the mixing in a room or other controlled environment having a temperature of (20 ± 2) °C and a relative humidity of not less than 50 %.

Place all the aggregate and then the cement in the dry mixing bowl (**5.11.4**) and mix for 30 s. Continue mixing and add the water during the next 30 s. Continue mixing for 60 s after all the water has been added. Stop the mixer and clean any adhering material from the paddle and sides with a scraper into the bowl, taking particular care to ensure that no unmixed materials remain at the bottom of the bowl, and complete these operations within 60 s. Cover the bowl with a damp cloth and allow to stand for 5 min.

Replace the bowl in the mixer and mix the mortar for a further 60 s.

15.3.8 Measurement of stiffening time

Immediately after completion of mixing of each mortar, determine the stiffening rate in accordance with EN 1015-9. Record the stiffening times for the duplicate test portions of the heated and unheated aggregates.

15.3.9 Compressive strength of hardened mortar

Prepare three 160 mm x 40 mm x 40 mm prisms by the procedure specified in EN 1015-11 from each mortar mix. Test the prisms for compressive strength at 28 days age. Record all 12 compressive strengths for the duplicate test portions of the heated and unheated aggregates. Determine the density of each prism on demoulding.

15.3.10 Calculation and expression of results

15.3.10.1 Stiffening time

Calculate to the nearest 15 min the change in stiffening time by subtracting the mean stiffening time of the heated aggregate mortars from the mean stiffening time of the unheated aggregate mortars.

NOTE: A negative result suggests that contaminators accelerated the setting of the mortar.

15.3.10.2 Compressive strength

Calculate to the nearest 1 % the relative compressive strength S % of the unheated aggregate mortar by the following equation:

$$S = A/B \times 100\ \%$$

where:

A is the mean compressive strength of the 6 unheated aggregate prisms in newtons per square millimetre;

B is the mean compressive strength of the 6 heated aggregate prisms in newtons per square millimetre.

NOTE: Organic contaminators may entrain or detrain air in mortar mixes. Entrained air affects compressive strength apart from any chemical effect which the contaminators may have on cement hydration. The presence of entrained air may be indicated by the masses of the unheated aggregate mortar prisms being significantly less than those of the heated aggregate mortar prisms.

15.3.11 Test report

The test report shall affirm that tests for contaminators which affect the setting and hardening of CEM I cement mortars were carried out in accordance with the requirements of EN 196-3 and whether or not a certificate of sampling is available. If available, a copy of the certificate of sampling shall be provided. The test report shall include the following additional information where applicable:

a) sample identification;
b) the change in stiffening time and relative strength of the unheated aggregate mortar.

16 Determination of water solubility

16.1 Principle

The aggregate test portion is extracted with twice its own mass of water in accordance with the procedure specified in **10.4**. After extraction, the recovered aggregate is dried and weighed.

16.2 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.

16.3 Preparation of test portion

Proceed as specified in **7.3**. Weigh the dried test portion to the nearest 0,1 g (m_{11}).

For fillers, reduce the test portion to (10 ± 0,2) g, weighed to the nearest 0,1 g.

16.4 Extraction of soluble components

After 24 h of extraction as specified in **10.4**, allow the major part of the solid settle. Filter away a maximum amount of the supernatant liquid on a pre-weighed medium-grade filter paper (**5.3.4**). Transfer quantitatively the settled aggregate, with the aid of a minimum amount of water, to a pre-weighed porcelain evaporating basin (**5.8.2**). Add filter paper and retained particles to the basin content. Dry and cool following the procedures specified in **6.4** and **6.5**. Weigh to the nearest 0,1 g and calculate the mass of aggregate by subtracting the masses of the basin and of the filter paper (m_{12}).

NOTE: For fillers, glass bottles should be used, with sufficient agitation to avoid any sedimentation.

16.5 Calculation and expression of results

Calculate the water solubility of the aggregate, expressed as *WS*, from the following equation:

$$WS = \frac{m_{11} - m_{12}}{m_{11}} \times 100 \quad \text{(in \%)}$$

where:

m_{11} is the mass of aggregate before extraction in grams;
m_{12} is the mass of aggregate after extraction in grams.

Record the result to 0,1 %.

17 Determination of loss on ignition

17.1 Principle

The loss on ignition is determined in an oxidizing atmosphere (air). By igniting in air at

(975 ± 25) °C, the carbon dioxide and the water not evaporated during drying are driven off as are any oxidizable volatile elements present.

NOTE: If aggregates contain non-volatile oxidizable constituents, as in the case of blast-furnace slags, the loss on ignition should be corrected in accordance with 7.4 of EN 196-2:1987.

17.2 Sampling and preparation of test portion

Proceed as specified in **11.2** and **11.3** to produce a test portion of mass (1 ± 0,05) g.

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 tared. Place the crucible in the electric furnace **(5.2.2)** controlled at (975 ± 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}).

NOTE: In the case of calcareous aggregates, heating to 975 °C has to proceed slowly, in order 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 equation:

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

where:

m_{13} is the mass of the test portion in grams;
m_{14} is the mass of ignited test portion in grams.

18 Determination of free lime in steel slag

18.1 General

Each of the methods specified in this clause determine the presence of free lime (CaO), which is potentially expansive, and hydrated lime ($Ca(OH)_2$) which is not expansive. To distinguish between these two forms of lime, extra tests are necessary, such as thermogravimetric or X-ray diffraction analysis.

18.2 Determination of free lime by complexometry (Reference method)

18.2.1 Principle

Free lime is extracted from a ground sample of aggregate by hot ethanediol. The content of calcium ions in the extract is subsequently determined by complexometric titration.

18.2.2 Sampling and preparation of test portion

Proceed as specified in **11.2** and **11.3**, but crushing the last 20 g until they all pass a 63 µm sieve and take approximately 0,5 g of this material as the test portion. Coarse iron fragments remaining on the sieve shall be removed.

18.2.3 Procedure

Weigh the test portion to the nearest 0,1 mg (m_{15}) and transfer into the conical flask (**5.12.1**) already containing a PTFE stirrer. Measure 50 ml of anhydrous ethanediol (**4.11.1**) and transfer into the flask. Seal the flask with the glass stopper and stir in a 70 °C waterbath for 30 min after reaching the temperature, using the magnetic stirrer at 300 rpm to 400 rpm. Then filter immediately through the sintered glass filter (**5.12.4**), which has a layer (approximately 4 mm to 5 mm) of firmly rammed filter paper pulp in ethanediol. Wash out the flask three times with a total of 50 ml propan-2-ol (**4.11.2**).

Acidify the clear filtrate, containing the dissolved free lime, with 10 ml of hydrochloric acid (1 + 1) (**4.11.4**) and rinse through with water into the measuring flask (**5.12.2**). Fill up to the mark and homogenize by shaking. According to the presumed content, transfer 50 ml or 100 ml to a glass beaker using a pipette. Add 10 drops of m-nitrophenol solution (**4.11.6**) and 10 drops of triethanolamine (**4.11.5**) (to sequester the Mn and Fe ions), and then neutralize with the 2 mol/l NaOH solution (**4.11.7**); dilute with water to approximately 500 ml and bring the pH-value to greater than 13 by adding about 10 ml of 2 mol/l NaOH solution. Add indicator (**4.11.8**) and titrate with EDTA solution (**4.11.9**) until the reddish mauve changes to blueish mauve. Determine the end point of titration by using the photoelectric titration equipment (**5.12.5**). A blank value shall always be determined from the ethanediol and the reagents.

18.2.4 Calculation and expression of results

Calculate the free lime content of the aggregate from the following equation:

$$\text{Free lime} = \frac{(V_{11} - V_{12}) \times F}{m_{15}} \quad (\text{in } \%)$$

where:

V_{11} is the volume of EDTA solution added (in millilitres)
V_{12} is the volume of EDTA solution added for the blank (in millilitres)
F is the EDTA solution factor in milligrams of CaO per millilitre; multiply by 0,5
in the case of pipetting 100 ml out of the flask (**5.12.2**)
m_{15} is the mass of the test portion (in grams).

The amount of free lime is rounded to the nearest 0,1 %.

18.3 Determination of free lime by conductometry (Alternative method)

18.3.1 Principle

Free lime is extracted from a ground sample of aggregate by hot ethanediol. The content of calcium ions in the extract is subsequently determined by conductance measurements.

18.3.2 Sampling and preparation of test portion

Proceed as specified in **18.2.2** and take (100 ± 0,1) mg of the passing 63 mm material as the test portion.

18.3.3 Procedure

Preheat 100 ml of ethanediol (**4.11.1**) to (80 ± 0,1) °C in the measuring vessel (see figure **2**) with the aid of a connected thermostat, stirring with a magnetic stirrer. Add the test portion to this solvent and introduce the measuring electrode.

NOTE: By measuring the conductance during extraction time, the dissolution of the free lime can be monitored directly.

After an extraction time of at least 10 min and when no further change in conductance occurs, the extraction process is completed. Then read off the final conductance value.

18.3.4 Evaluation and expression of results

Carry out the conversion of the measured conductivity into free lime content using a calibration graph (see figure **3**). Express the mass percentage of free lime to the nearest 0,1 %.

18.4 Determination of free lime by acidimetry (Alternative method)

18.4.1 Principle

Free lime is extracted from a ground sample of aggregate by boiling in ethyl acetoacetate (Franke method); the extract is titrated with a standard 0,2 mol/l hydrochloric acid solution.

18.4.2 Sampling and preparation of test portion

Proceed as specified in **18.2.2** and take approximately 1 g of the passing 63 mm material as the test portion.

18.4.3 Procedure

Measure 70 ml of the prepared solvent solution (**4.13.5**) containing ethyl acetoacetate and 2-methylpropan-1-ol in the proportion of 3 to 20, and transfer into the Erlenmeyer flask (**5.14.1**). Weigh the test portion to the nearest 0,1 mg (m_{16}) and transfer into the flask.

Adjust the flask in position to the water-cooled condenser, fitted with the upper adapter tube containing the sodium hydroxide (**4.13.8**) and the molecular sieve (**5.14.3**); reflux at boiling temperature, stirring on the hot plate (**5.2.7**) for 3 h. Remove the hot plate, allow to cool, then filter under vacuum through the glass microfibre filter (**5.14.4**), receiving the filtrate in a second flask. Wash the first flask and residue with 50 ml of 2-methylpropan-l-ol (**4.13.2**), using a stirring rod fitted with a rubber policeman to guide the flow.

Add 10 to 12 drops of the indicator solution (**4.13.6**) to the filtrate and titrate with the standard 0,2 mol/l hydrochloric acid solution (**4.13.7**) to a distinct reddish tinge.

NOTE: If the titration is conducted by means of a recording pH meter, the filtration of the extract is not needed.

18.4.4 Calculation and expression of results

Calculate the free lime content of the aggregate from the following equation:

$$\text{Free lime} = k/1000 \times V_{12}/m_{16} \times 100 \text{ (in \%)}$$

where:

V_{12} is the volume of hydrochloric acid added (in millilitres);
m_{16} is the mass of the test portion (in grams);
k is the factor defined in **4.13.7**, representing the number of milligrams of free CaO per millilitre of standardized hydrochloric acid solution.

19 Determination of unsoundness of blast-furnace and steel slags

19.1 Determination of dicalcium silicate disintegration of air-cooled blast-furnace slag

19.1.1 General

This clause specifies the method of determination of the susceptibility to disintegration of crushed blast-furnace lump slag resulting from the inversion of the metastable ß form of the dicalcium silicate to the γ form. This phenomenon is sometimes called improperly 'lime disintegration'.

19.1.2 Principle

Broken slag surfaces fluoresce under ultra-violet light in the range of visible light. The aspect and colour of fluorescence enable the detection of slags suspect with respect to silicate disintegration.

19.1.3 Sampling

Proceed as specified in **11.2**.

19.1.4 Preparation of test portion

Reduce the laboratory sample to a test portion of at least 30 lumps, then wash and dry the test portion, then split each lump to obtain freshly broken surfaces.

19.1.5 Procedure

Carry out the dicalcium silicate disintegration test under ultra-violet light (**5.15.1**).

19.1.6 Expression of results

Record the observations made on the appearance of freshly broken surfaces. Slags which exhibit numerous or clustered large and small bright spots of a yellow, bronze or a cinnamon colour on a violet background shall be recorded as suspect with respect to disintegration.

Slags with a uniform shine in various shades of violet and those exhibiting bright spots in a limited number only and uniformly distributed, shall be deemed sound.

19.2 Determination of iron disintegration of air-cooled blast-furnace slag

19.2.1 General

This clause specifies the method of determination of the susceptibility to disintegration of crushed blast-furnace slag, resulting from the hydrolysis of iron- and manganese sulfides.

19.2.2 Principle

The iron disintegration occurring by ageing in a humid atmosphere or in rain, but more rapidly under water, is observed by examining the behaviour of pieces of slag which have been immersed in water.

19.2.3 Sampling

Proceed as specified in **11.2**.

19.2.4 Procedure

Place 30 pieces of slag, with a nominal size between 40 mm and 150 mm, in water at (20 ± 2) °C for two days.

19.2.5 Expression of results

Record any cracking or disintegration. If no pieces disintegrate or crack, the sample shall be regarded as having passed the test. If one or two pieces disintegrate or crack, the test shall be repeated with a further 30 test pieces. If any pieces disintegrate or crack in the second test the sample shall be regarded as having failed the test.

19.3 Determination of the expansion of steel slag

19.3.1 General

This clause specifies the method of determination of the susceptibility to expansion of crushed steel slag, resulting from the late hydration of dead burned free lime and/or free magnesium oxide.

19.3.2 Principle

A compacted slag specimen, combined from known grain sizes, is subjected to a flow of steam at 100 °C in a steam unit at ambient pressure. By this means, the necessary moisture for reaction with the free lime and free magnesium oxide is continuously conveyed to the test specimen. Any change in the volume caused by this reaction is read off from a dial test indicator directly at the top of the specimen. The increase in volume is given as the result, calculated in % volume in relation to the original volume of the compressed slag specimen.

19.3.3 Sampling

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

19.3.4 Preparation and compaction of the specimens

The laboratory sample taken shall be dried immediately in the laboratory at (110 ± 5) °C until a constant mass (**6.5**) is reached.

For the steam test use 0 mm to 22 mm test portions of dried mineral mixtures of slag, which have been combined according to the Fuller parabola. The proportions of mass for the individual grain size classes are given table **3**:

Table 3: Proportions of mass per grain size class

Sizes mm	Percentage in mass
0 to 0,5	15
0,5 to 2	15
2 to 5,6	19
5,6 to 8	10
8 to 11,2	11
11,2 to 16	15
16 to 22	15
Total	100

The individual grain size classes shall be taken from the crushed aggregates.

The sample reduction shall be carried out in accordance with the procedures specified in prEN 932-2.

The expansion shall be determined on at least two individual test specimens. Each individual test portion shall be combined separately with the above-mentioned size distribution. The amount of material required for each individual sample is 4,5 kg. In addition a sample shall be combined for the determination of the apparent density, in accordance with prEN 1097-6.

Cover the perforated base of the test cylinder with a circular filter (fabric mat) and, using a laboratory scoop, transfer the prepared test portion to the cylinder (**5.16.1**) illustrated in figure **5**. Then dynamically compact the dry specimen on the vibrating table (**5.16.5**) for 6 min at a frequency of (48 ± 3) Hz (amplitude ± 1,5 mm) and a static load of 0,035 N/mm², produced for example by hydraulic pressure. Under these test conditions, a practical void content of between 20 % volume and 25 % volume remains in the slag specimen, values conforming to the practice. Alternatively, the sample can be compacted by any other method capable of achieving the same degree of compaction, such as a Proctor hammer or a hand-held power hammer. If water is added to the sample to facilitate compaction, testing shall commence within 24 h of the completion of the compaction process.

After compaction, determine the volume of the slag specimen V_S, which is the difference between the volume of the cylinder V_C and the volume of air V_A between the slag specimen and the top edge of the cylinder. Calculate V_C and V_A from height measurements with the sounding rod (**5.16.6**) and taking the average of four readings on the extremity of two at right angles crossing diameters

Finally, cover over the surface of the slag specimen with a circular filter (fabric mat) and then with a layer of glass beads (**5.16.3**) which have been lubricated before the test with silicone oil in order to reduce friction between the individual beads. The total mass of the layer of beads is (1,5 ± 0,01) kg; distribute the glass beads evenly within the test cylinder so that they give a level surface.

NOTE: 1,5 g of silicone oil is sufficient for the batch of 1,5 kg of beads.

Repeat the lubrication of the glass beads after each steam test. As lime is deposited on the glass beads during the steam test, it is furthermore necessary to clean off the lime after every fourth steam test with dilute hydrochloric acid.

19.3.5 Steam test procedure

After the slag test specimen has been covered with the layer of glass beads, attach the test cylinder to the steam equipment and fit the heating jacket to the outer wall of the test cylinder. Then lay the perforated plate, the weighting cross and the loading weight on top of the beads. Fix the dial indicator or displacement meter, which registers the lifting movements of the specimen surface, to a rigid support on the steam unit. After switching on the heating jacket and the steam unit, the slag test specimen then starts heating up with an expansion of its volume. In order not to register the lifting movements associated with this, do not begin recording the displacement until steam is passing freely through the sample.

The testing time in the steam test lasts altogether 24 h for LD-slags and 168 h (7 days) for electric arc furnace and open hearth furnace slags (steel- making slags). At the end of this period the rise in the test specimen surface is read off and calculated in % volume in relation to the original volume (see **19.3.6**).

The water container in the steam unit is of a size such that the steam test can be performed over a period of 24 h. When it is necessary to top up with water, care shall be taken to prevent a drop in temperature which would interrupt the production of steam.

NOTE: In many cases, it is useful to record the development of the increase in volume as dependent on time. As the movements at the beginning of the steam test occur sharply, it is recommended that the movements be read off at intervals of 15 min. After 4 h the interval can be extended to 60 min. If the increase in volume is taken as a function of the time, a detailed interpretation of the test results can be undertaken by means of the graph (starting climb, asymptotic approach to a limit value).

19.3.6 Calculation and expression of results

Calculate the volume of the slag test portion V_S, before the steam test, from the following equation:

$$V_S = V_C - V_A$$

where:

V_S is the volume of the slag test specimen after compaction in the test cylinder (in cubic centimetres);

V_C is the volume of the cylinder (in cubic centimetres);

V_A is the volume of air between the slag specimen and the top edge of the cylinder (in cubic centimetres).

V_C and V_A are calculated from the height measurements with the sounding rod and the diameter of the cylinder (210 mm)

After compaction determine the bulk dry density and the void content of the compacted mixture as follows:

$$r_M = \frac{100\,W}{V_S\,(100 + w)} \quad \text{(in megagrams per cubic metre) and}$$

$$V_M = (1 - r_M/r) \times 100 \quad \text{(in \% volume)}$$

where:

r_M is the bulk density of the compressed mixture,(in megagrams per cubic centimetre);
W is the mass of the compressed mixture,(in grams);
V_M is the void content of the compressed mixture, (in % volume);
V_S is the volume of the slag test specimen after compaction in the test cylinder (in cubic centimetres);
r is the particle density of the slag, determined as specified in prEN 1097-6 (in megagrams per cubic metre);
w is the water content of the sample (in percentage mass).

After completing the test, calculate the expansion in volume % from the rise of the specimen read off the dial indicator or meter display, and from the internal diameter of the test cylinder (210 mm) from the following equation:

$$\text{Expansion} = \frac{\text{p} \times h \times d^2}{4 \times V_S} \times 100 \quad \text{(in \% volume)}$$

where:

h is the rise of the specimen after the steam test (in millimetres);
d is the internal diameter of the test cylinder (210 mm).

Record the result as the arithmetical mean of the expansion of the 2 test specimens, rounded to the nearest 0,1 % volume.

Annex A (informative)

Precision

A.1 Symbols

r_1 is the repeatability limit as defined in prEN 932-6.

R_1 is the reproducibility limit as defined in prEN 932-6.

X is the average of the test results.

A.2 Determination of water-soluble chloride salts using the Volhard method (Reference method) (see clause 7)

The precision of the determination of water soluble chlorides is stated (in terms of the chloride ion content, as a percentage by mass of the aggregate) as:

$$r_1 = 0{,}0004 + 0{,}029\,X \text{ and } R_1 = 0{,}0006 + 0{,}124\,X$$

A.3 Determination of water-soluble chloride salts by potentiometry (Alternative method) (see clause 8)

The standard deviation for repeatability r is 0,001 %.
The standard deviation for reproducibility R is 0,003 %.

A.4 Determination of total sulfur content (Reference method) (see clause 11)

The precision of the determination of total sulfur content is stated (in terms of the sulfur content, as a percentage by mass of the aggregate) as:

$$r_1 = 0{,}017 + 0{,}081\,X \text{ and } R_1 = 0{,}062 + 0{,}204\,X$$

A.5 Determination of acid soluble sulfates (Reference method) (see clause 12)

The precision of the determination of acid soluble sulfate content is stated (as the percentage SO_3 by mass of the aggregate) as:

$$r_1 = 0{,}021 + 0{,}200\,X \text{ and } R_1 = 0{,}000 + 0{,}812\,X$$

Annex B (informative)

Bibliography

List of the principal documents which have served as references in the preparation of the standard.

prEN 932-6	Tests for general properties of aggregates Part 6: Definitions of repeatability and reproducibility
BS 812	Testing aggregates: Part 117:1988 Method for determination of water-soluble chloride salts Part 118:1988 Method for determination of sulfate content Section 122.1 Organic contaminators which influence the setting and hardening of Portland cement mortars (draft) Section 122.2 Lightweight contaminators which may disfigure concrete and mortar (draft)
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