BRITISH STANDARD

BS EN 14629:2007

Products and systems for the protection and repair of concrete structures — Test methods — Determination of chloride content in hardened concrete

The European Standard EN 14629:2007 has the status of a British Standard

ICS 91.100.30

National foreword

This British Standard was published by BSI. It is the UK implementation of EN 14629:2007.

The UK participation in its preparation was entrusted by Technical Committee B/517, Concrete, to Subcommittee B/517/8, Protection and repair of concrete structures.

A list of organizations represented on B/517/8 can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was
published under the authority
of the Standards Policy and
Strategy Committee
on 30 April 2007

ISBN 978 0 580 50528 7

Amendments issued since publication

Amd. No.	Date	Comments

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14629

March 2007

ICS 91.100.30

English Version

Products and systems for the protection and repair of concrete structures - Test methods - Determination of chloride content in hardened concrete

Produits et systèmes pour la protection et la réparation des structures en béton - Méthodes d'essais - Mesurage du taux de chlorure d'un béton durci

Produckte und Systeme für den Schutz und die Instandsetzung von Betontragwerken - Prüfverfahren - Bestimmung des Chloridgehaltes von Festbeton

This European Standard was approved by CEN on 7 September 2006.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN Management Centre 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 CEN Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 14629:2007: E

Contents

Page

Foreword

This document (EN 14629:2007) has been prepared by Technical Committee CEN/TC 104 “Concrete and related products”, the secretariat of which is held by DIN.

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 2007, and conflicting national standards shall be withdrawn at the latest by September 2007.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

It has been prepared by sub-committee 8 "Products and systems for the protection and repair of concrete structures" (Secretariat AFNOR).

This European Standard is one of a series dealing with products and systems for the protection and repair of concrete structures. It describes a method for determining the chloride content of hardened concrete.

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

Introduction

Steel reinforcement in concrete may be at risk of corrosion if the concrete is contaminated by chlorides.

ENV 1504-9 defines the principles for protection and repair of concrete structures which have suffered or may suffer damage or deterioration and gives guidance on the selection of products and systems which are appropriate for this intended use. EN 1504-10 requires that the concentration of chlorides be considered when decisions about the removal of concrete are made.

To establish whether there is a risk of corrosion of the steel reinforcement due to a higher chloride content within the concrete than the critical threshold value, the chloride content within the concrete cover and especially at the surface of the steel reinforcement should be determined. Assessment may be made in the field using a variety of rapid test methods, which are not included in this standard.

For accurate determination of chloride content, concrete samples need to be taken from the structure, prepared for chemical analysis and analysed. For chemical analysis of the concrete with respect to the chloride content either Volhard’s method or potentiometric titration are used as the reference methods.

The chloride content may be expressed as a percentage of chloride by mass of cement or of concrete. The chloride content by mass of cement may be derived using either an assumed value for the cement content of the concrete or a value which has been determined by chemical analysis or from construction records.

1 Scope

This standard describes two methods for the determination of the total (free and bound) acid soluble chloride content of hardened concrete or mortar. This information is intended for use in estimating the risk of chloride induced corrosion of the steel reinforcement. It may be used on samples of powder obtained either by drilling or from cores or fragments removed from concrete structures or on other appropriate laboratory specimens.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Materials and apparatus

3.1 Chemicals

- Deionised water with an electrical conductivity less than 2µS/cm;
- Nitric acid (5 mol/l);
- Silver nitrate solution (0,02 mol/l);
- Ammonium thiocyanate (NH_4SCN) solution (0,1 mol/l);
- Ammonium ferric sulfate indicator solution (100 ml of a cold saturated solution of $NH_4Fe(SO_4)_2$ and 10 ml diluted nitric acid);
- 3,5,5-trimethylhexanol.

NOTE For the potentiometric method, additional reagents may be required as specified by the electrode manufacturer to ensure the proper functioning of the electrode. These will be specified in the manufacturer's instructions for the electrode.

3.2 Apparatus

- Crushing and grinding equipment (as required to prepare samples);
- 1,18 mm sieve or smaller;
- ventilated oven, controlled to maintain a temperature of (105 ± 5) °C;
- balance capable of weighing up to 5 g to an accuracy of 1 mg;
- desiccator;
- burette, accurate to 0,05 ml;
- 250 ml glass beaker;
- magnetic stirrer;

- heat source;
- vacuum filtering facility (Buchner funnel, filtration flask, medium-textured filter paper);
- 250 ml volumetric flask;
- pipettes accurate to 0,1 ml;
- if required, potentiometric titration facility (eg. Ag/AgCl electrode or similar, high resistance mV-meter, burette accurate to 0,05 ml or automatic titration equipment).

All volumetric glassware shall be of class A accuracy as defined in ISO 384.

4 Test procedure

4.1 Sampling

4.1.1 Sampling plan

Sampling shall be in accordance with a plan prepared for the assessment or repair works by a suitably qualified person. When specifying the locations from which concrete is to be removed, specific care shall be taken of the load bearing capacity and reinforcement of the elements to be sampled. The plan shall take into account:

- the size, form, location, orientation, age and structural design of the structure;
- the results of visual surveys and any other available information about possible deterioration;
- the aggregate size and the heterogeneous nature of the concrete;
- exposure conditions, eg. salt water splash zones.

On site checks shall be carried out to ensure that the concrete to be sampled is representative of the elements to be investigated and to confirm the location of reinforcement, which should not be damaged during sampling.

The plan shall specify the approximate number, location and depth of samples, including the depth increments. Typically increments do not exceed 25 mm, and it is usual to discard the surface layer.

NOTE All holes or damage to the structure from sampling should be repaired or protected immediately to prevent further deterioration.

4.1.2 Collecting powder samples

When drilling for powder samples the diameter of the drilling tool and therefore the number of drill holes required to obtain a sufficient sample size is determined by the maximum aggregate size. The powder is collected in separate depth increments, as specified in the sampling plan. A minimum sample size of approximately 1 g is required.

NOTE A drill diameter of 20 mm and two holes is normally sufficient to provide 1 g of powder per mm depth of increment. For concrete containing aggregates of maximum size of 20 mm a 20 mm drill should be used. A sample of at least 20 g is normally collected to ensure that it is representative of all of the concrete constituents and not just the aggregate. For larger aggregate sizes a larger drill diameter and sample size should be used.

4.1.3 Taking core samples

A core diameter of 30 mm to 50 mm, depending on the maximum aggregate size, is usually recommended. Larger sizes may be required for large aggregate sizes. Cores shall be marked to indicate their location and orientation with respect to the original concrete surface.

For determination of chloride profiles related to the distance from the concrete surface, the cores may be cut into slices as specified in the sampling plan, without using cooling fluid, or may be ground in increments to obtain powder samples.

4.2 Grinding

Where the sample requires grinding, it shall be dried in an oven to constant weight at (105 ± 5) °C and then allowed to cool to room temperature, for example in a desiccator. When cool it shall be ground to a fine powder to pass a 1,18 mm sieve or smaller, and then homogenised.

4.3 Chemical analysis

4.3.1 General requirements

The mass of samples shall be given in grams to the nearest 0,001 g and volumes given in millilitres to the nearest 0,05 ml.

Approximately one sample out of every 20 samples (or one sample from every batch if less than 20 samples in the batch) shall be an interlaboratory standard concrete dust specimen of known chloride content. Occasional duplicate analyses, in which the same sample is analysed twice and the results compared, shall also be carried out to confirm the accuracy of the analysis.

NOTE In selecting samples for duplicate analyses note should be taken of the results obtained, in particular of any results which fall outside general trends such as a reduction of chloride concentration with sample depth.

4.3.2 Dissolving chlorides

Between 1 g and 5 g of concrete powder shall be weighed and placed in a 250 ml beaker, wetted with 50 ml water, and 10 ml of 5 mol/l nitric acid added, followed by 50 ml hot water.

NOTE 1 Adding concentrated acid allows the lab to use a dispenser. This saves time and avoids cross contamination. The addition of hot water quickens boiling and avoids spitting.

The mixture shall be heated until boiling and boiled for at least 3 min, stirring continuously.

If necessary the mixture shall be filtered immediately using medium-textured paper, washing the beaker, the stirrer and the residue on the filter.

NOTE 2 It is not necessary to filter the solution for potentiometric titration.

4.3.3 Blank solution

Carry out the same procedure with no concrete test portion.

4.3.4 Determination of chloride content

4.3.4.1 Volhard's Method (Method A)

Add 5 ml of silver nitrate solution by pipette into the test solution and stir vigorously to precipitate the chloride.

If the chloride content is high, and a large amount of precipitate is formed, a floculating agent such as 3,5,5-trimethylhexanol may be added. It can be added after the silver nitrate, so the chloride level can be judged from the amount of precipitate formed.

NOTE The smell of 3,5,5-trimethylhexanol is overpowering and it is recommended to use it as little as possible.

Add 5 drops of indicator solution and titrate with the ammonium thiocyanate solution one drop at a time, while continually agitating the solution until the faint reddish-brown coloration no longer disappears. Record the volume V_1 of solution used in the titration.

An early end-point, at less than 1 ml of ammonium thiocyanate addition, indicates that the test solution demands more silver nitrate solution than the 5,0 ml already added. In this case a further 5 ml of $AgNO_3$ shall be added and the titration continued until the end point is reached a second time. Record the volume V_1 of solution used to reach the second end point. This procedure may be repeated up to three times. If the sample contains too much chloride then repeat from the beginning (4.3.2) with a smaller sample mass.

Calculate the chloride content as percent of chloride ion by mass of sample using the following formula:

$$CC = 3.545 * f * (V_2 - V_1) / m$$

V_1 volume of the ammonium thiocyanate solution used in the titration [ml];

V_2 volume of the ammonium thiocyanate solution used in the blank titration [ml];

m mass of the concrete sample [g];

f molarity of silver nitrate solution.

4.3.4.2 Potentiometric titration (Method B)

The chloride content is determined with 0,1 M silver nitrate solution in a potentiometric titration. Consumption of silver nitrate solution (V_3) in the titration is noted.

The first derivative method is to be used. First derivative is a technique of adding small aliquots of titrant to the sample, recording the potential changes, and applying a first derivative analysis to the data, from which the endpoint is calculated. The technique assumes that the change in mV reading per volume of titrant added will be greatest at the endpoint.

NOTE Some instruments work better when a small amount of chloride (equivalent to appr. 0,1 ml of silver nitrate consumption) is added to the sample before digestion. The manufacturer's instructions should be followed.

Carry out the same procedure with a blank solution (with added chloride as for samples if appropriate) and record the volume (V_4) of silver nitrate solution used in the blank titration.

Calculate the chloride content of the concrete as percent of chloride ion by mass of sample using the following formula:

$$CC = 3.545 * f * (V_4 – V_3) / m$$

V_3 volume of the silver nitrate solution used in the titration [ml];

V_4 volume of silver nitrate solution used in the blank titration [ml];

m mass of the concrete sample [g];

f molarity of silver nitrate solution.

5 Test report

The test report on the determination of the chloride content should include the following information:

a) client's name;

b) date of test;

c) unique sample identification no (and locations of all samples if known);

d) mass, size and type of specimen used (ie. powder, core or fragment), for dust samples a mass range including lowest and highest mass is satisfactory;

e) chloride content as percent of chloride ion by mass of sample of concrete;

f) chloride content of the interlaboratory standard sample and the accuracy;

g) if requested, chloride content by mass of cement - and how calculated;

NOTE If requested, chloride content by mass of cement may be calculated, provided that the cement content of the concrete is known; in such a case, the method of establishing the cement content should be given. Otherwise, an estimate based on an assumed value of cement content may be made, but the basis of the assumption should be clearly stated. If the cement content is assumed a value of 14 % cement content is often used for the calculations for a typical normal density concrete. A concrete containing 350 kg/m^3 of cement with a density of 2 400kg/m^3 has a cement content of about 14 % by weight.

h) method of determination (Volhard or Potentiometric);

i) reference to this European Standard.

Any available information on e.g. the mix design and age of the concrete should be reported.

Bibliography

[1] ENV 1504-9, *Products and systems for the protection and repair of concrete structures — Definitions, requirements, quality control, evaluation of conformity — Part 9: General principles for the use of products and systems*.

[2] EN 1504-10, *Products and systems for the protection and repair of concrete structures — Definitions, requirements, quality control, evaluation of conformity — Part 10: Site application of products and systems and quality control of works*

blank

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: +44 (0)20 8996 9000. Fax: +44 (0)20 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: +44 (0)20 8996 9001. Fax: +44 (0)20 8996 7001. Email: orders@bsi-global.com. Standards are also available from the BSI website at http://www.bsi-global.com.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: +44 (0)20 8996 7111. Fax: +44 (0)20 8996 7048. Email: info@bsi-global.com.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: +44 (0)20 8996 7002. Fax: +44 (0)20 8996 7001. Email: membership@bsi-global.com.

Information regarding online access to British Standards via British Standards Online can be found at http://www.bsi-global.com/bsonline.

Further information about BSI is available on the BSI website at http://www.bsi-global.com.

Copyright

Details and advice can be obtained from the Copyright & Licensing Manager. Tel: +44 (0)20 8996 7070. Fax: +44 (0)20 8996 7553. Email: copyright@bsi-global.com.

BSI
389 Chiswick High Road
London
W4 4AL