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Concrete — Release of regulated dangerous substances into soil, groundwater and surface water — Test method for new or unapproved constituents of concrete and for production concretes

ICS 91.100.30

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National foreword

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English Version

Concrete - Release of regulated dangerous substances into soil, groundwater and surface water - Test method for new or unapproved constituents of concrete and for production concretes

Béton - Relargage de substances dangereuses réglementées dans les sols, les eaux souterraines et les eaux de surface - Méthode d'essai des constituants du béton, nouveaux ou non-approuvés, et des formules de béton

Beton - Freisetzung regulierter gefährlicher Stoffe in den Boden, das Grundwasser und das Oberflächenwasser - Testmethode für neue oder noch nicht zugelassene Bestandteile von Beton und für Betone

This Technical Report was approved by CEN on 1 October 2007. It has been drawn up by the Technical Committee CEN/TC 51.

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Foreword

This document (CEN/TR 15678:2008) has been prepared by Technical Committee CEN/TC 51 “Cement and buliding limes”, the secretariat of which is held by NBN.

It describes test methods, that when completed, will produce eluates for the assessment of inorganic and organic substances potentially released from either the constituents of concretes (tested within hardened reference concretes) or from production concretes (or test pieces representative of production concretes) whether presented in the pre-hardened/precast state, fresh wet state or pre-packaged.

This document is currently incomplete. This document does not include the extraction procedure necessary to produce the eluates because extraction procedures will be developed within a programme of horizontal test method standardisation under a mandate given to CEN by the European Commission and the European Free Trade Association in order to support essential requirements of EU Directives. The necessary programme of work is being undertaken in CEN/TC 351 Construction products – Assessment of release of dangerous substances.

Annex A, which is normative, describes the testing for release of (regulated) dangerous substances from pre-hardened concrete products formed in the factory.

Annex B, which is informative, describes the testing for release of (regulated) dangerous substances from hardened test pieces representative of fresh wet concretes or pre-packaged concretes.

Annex C, which is informative, describes the principles of laboratory concrete mix design to be applied for the assessment of new/unapproved constituents of concrete.

Introduction

Regulatory background

In March 2005 the Commission Services of the European Union published mandate M/366 “Development of horizontal standardised assessment methods for harmonised approaches relating to (regulated) dangerous substances under the Construction Products Directive (CPD)”. M/366 deals with the subject of emissions or release of (regulated) dangerous substances from construction products, as defined in the CPD, which may have harmful impacts on human health and the environment in relation to essential requirement No. 3 (ER 3), Health, hygiene and the environment, of the CPD. The mandate is intended to provide harmonised European measurement/test method standards that are needed in order to remove technical barriers to trade and bring about the “approximation” of laws, regulations and administrative provisions of the Member States. The measurement/test standards should provide results that can be expressed in performance terms and be suitable for addressing the emission or release of (regulated) dangerous substances within provisions in harmonised European Technical Specifications (ETS).

For ease of assigning the appropriate environments or exposure scenarios to individual products, the environment is notionally divided into two distinct environmental compartments: indoor air and soil, groundwater and surface water. In this context, it is important to recognize that essential requirement No. 3 only covers the potential effects of construction on the health of occupants and neighbours of construction works and the environment immediately surrounding the works. In life-cycle terms, it covers only the service life of a product and, therefore, does not cover the construction phase or end-of-use/disposal.

These distinct environmental compartments are necessarily associated with exposure-specific test methodologies, the emission of volatile, semi-volatile or other substances into indoor air being physico-chemically, kinetically and thermodynamically distinct from release into aqueous environments. The test methods described herein address only the potential release of substances into soil, groundwater and surface water, forms of aqueous exposure which, given the focus on service-life, could be described as the natural environment. Assessment of emission of substances into indoor air may or may not be relevant for the cementitious products identified in this CEN TR. However, if it emerges that the regulatory regime requires that this aspect of performance be addressed, then the European cement and concrete sector, via its standardization committees, will involve itself in appropriate work items.

At the time of drafting this CEN TR, spring 2007, there are a number of uncertainties about the regulatory regime that will eventually govern the assessment of emission/release of dangerous substances from construction products within Europe. In particular, it is unclear which products and/or materials will be subject to any part of the emerging regulatory regime. In the case of cement-containing products or materials such as concrete there is additional uncertainty because, with the exception of mixing water, all the constituents of concrete are construction products mandated in their own right under the Construction Products Directive (CPD). However, the constituents of concrete do not come into direct contact with either soil, groundwater or surface water and this could be taken to infer that they should not be subject to any regulatory provisions except that some (i.e. those not already standardised under national standards or European Technical Specifications) are subject to assessment within some Member States' existing environmental regulations. Furthermore, some types of concrete, for example fresh wet concretes, have not been mandated as construction products under the CPD and therefore might be considered to be outside any European regulatory regime based upon it. The complicating factor for these materials, though, is that they are subject to some EU Member States' existing regulations and may, in consequence, need to be assessed under a European regulatory regime.

It is also unclear whether the assessment and classification framework under development for use in the European regulatory regime will eventually be adopted. Currently, however, there are indications that the framework will include three distinct elements:

a 'deemed to satisfy' classification scheme for assessing some products on the basis of existing, generally accepted knowledge, currently known as 'without testing (WT)';

a classification scheme known as 'without further testing (WFT)' but based on initial testing using agreed European test methods may also form part of the regime;

and for use in those cases where the above classifications are either inapplicable or where products cannot achieve either classification because they can emit or release substances in amounts in excess of the classification criteria, there would be a need to carry out 'further testing (FT)' in the form of routine or conformity testing, again using agreed European test methods called up from appropriate provisions placed in harmonised European Technical Specifications (e.g. harmonised European product standards and European Technical Approvals). However, it is clear that the eventual European regulatory regime will neither establish nor include any pan-European assessment criteria for assessing emission or release under conditions of 'further testing (FT)'. Assessment under FT conditions will be a matter of comparing results obtained using European test methods with the appropriate, and possibly appropriately modified, limiting criteria in EU Member States' existing national regulations.

Given the regulatory uncertainties outlined above, this CEN TR has taken the position that its scope must initially include:

all types of concrete [pre-hardened/precast, fresh wet (ready-mixed and site-mixed) and pre-packaged);

and all the constituents of concrete, with the exception of mixing water;

so as to cover the eventuality that any of these, whether mandated construction products or not, may need to be assessed for the purposes of conformity with Essential Requirement 3 of the Construction Products Directive because any can come into either direct or indirect (constituents of concrete) contact with soil, groundwater or surface water. However, those products that have been mandated as construction products in their own right under the CPD are dealt with in normative provisions in this CEN TR whereas those that have not been mandated are dealt with in informative provisions.

Overall scope of products and test methods

This CEN TR describes three distinct test methods but all use the same extraction procedure. The first will permit the constituents (e.g. cement, additions, aggregates, admixtures, fibres etc) of concrete that have not been officially classified as WT products (i.e. without testing to a European standard test method being required) to be assessed against any European or national requirements for release of (regulated) dangerous substances from hardened test pieces of concrete into soil, groundwater or surface water.

The second method, in normative Annex A, is designed to permit factory made pre-hardened concretes, or test pieces representative of factory made concretes, which have not been officially classified as WT products, to be assessed against any European or national requirements for release of (regulated) dangerous substances into soil, groundwater or surface water.

The third method, in informative Annex B, is designed to permit concretes sampled in the fresh wet state or pre-packaged (and also not officially classified as WT products) to be assessed in the hardened state, against any European or national requirements for release of (regulated) dangerous substances into soil, groundwater or surface water.

Construction products, whether constituents of concrete or concrete itself, that have been officially classified as WT products by way of authorised procedures will not require to be tested by the methods described herein. In consequence, the overall scope is directed to the testing of new or previously unapproved constituents of concretes, or to production concretes where a requirement to test the end-use product has arisen.

Constituents - general description of the method of test

The first method describes how to determine the release of (regulated) dangerous substances from a new/unapproved constituent of concrete. The method is comparative in that results obtained from a laboratory prepared reference concrete which does not include the constituent under test (control mix) are subtracted from the results obtained from a reference concrete that incorporates the test constituent by either substitution or addition (test mix).

A new/unapproved constituent for use in concrete can be incorporated into a reference concrete in three different ways, either by:

- *substitution* (partial or full) for a reference constituent of the same type and which reference constituent has either been classified as WT or has otherwise demonstrated its fitness for intended use, as in the case of: factory-made cements, aggregates, type I additions;
- *substitution* (partial) for a reference constituent of a different type and which reference constituent has either been classified as WT or has otherwise demonstrated its fitness for intended use, as in the case of: type I or type II additions;
- or by *addition* to the reference concrete, as in the case of: admixtures, polymer modifiers and fibres.

As a principle, the method seeks to isolate, as far as is practicable, the contribution to the release of substances from the unapproved constituent i.e. the constituent under test, from the contributions from the other constituents that make up the reference concrete. However, complete isolation of contributions to release, one constituent from another, is not possible for all types of constituents. For example, if *substitution* by a type II addition or *addition* by a chemical admixture leads via a synergistic effect (e.g. by a micro-structural modification) to a reduction in permeability of a test mix in comparison with its companion control mix, then complete isolation will not have been achieved. Such synergies, however, occur in concretes as used in the field, therefore, the overall effects on release from the relevant hardened test mixes will also tend to reflect reality.

In general, in the case where an unapproved constituent replaces a reference constituent by *substitution*, the assessment is made by subtracting, in whole or in part (depending on the type of constituent and the level of substitution), the results for release of substances obtained for the reference concrete (i.e. the control mix) from those obtained for the test mix in which the unapproved constituent is present. The exception to this rule occurs in the specific case where the unapproved constituent is a factory made cement. In this particular case the design of the method is such that there will be no contributions from other constituents to be subtracted.

Similarly, in the case where an unapproved constituent is *added* to a reference concrete, the assessment is made after subtracting any contributions to release of substances from the reference concrete in which the constituent under test is absent (i.e. the control mix) from any contributions from the test mix to which the constituent has been added.

Production concretes - general description of the method of test

The second and third methods, in normative Annex A and informative Annex B, respectively, utilise the same test conditions for assessing release of (regulated) dangerous substances but start from the sampling of production concretes. In cases where products are supplied to the market as formed in the factory, they may be sampled as either finished items (monoliths) or as proxy samples of the fresh wet material used in their production. Where materials are supplied in a formless state, samples are taken from the fresh wet materials. Where products are supplied pre-packaged, each sample is a representative whole bag of product.

In the case of products sampled in the fully formed (monolithic) state, test pieces are prepared by either sawing or coring regular shaped test pieces of appropriate dimensions from the factory-made item. Whereas, in the case of samples of fresh wet material and pre-packaged products, test pieces are prepared as standard-sized moulded test pieces.

Moulded test pieces are cured under different conditions depending on the product type from which they are taken. In the case of proxy samples taken from fresh wet material used in the production of factory made

items, these are cured under conditions that are representative of those used in the manufacture of the item. In the case of test pieces prepared from pre-packaged products and fresh wet materials as supplied to the market, these are cured for periods specified in specifications or regulations appropriate to the material type. Subsequent to curing, test pieces, representing production of concrete, are subject to the same extraction conditions as those used for testing individual constituents in reference mixes of concrete.

However, in the case of production materials, there is no place for a reference concrete in the test method because it is the overall release of substances from the sample taken from production that is to be assessed against either generic limit (e.g. threshold) values or classes of performance appropriate to concrete, rather than, as in the first method, limit values or performance classes appropriate to individual constituents.

Extraction conditions

The extraction procedure to be adopted by the methods described herein will be the horizontal monolithic bulk diffusion method developed by CEN/TC 351 *Construction products – Assessment of release of dangerous substances*, established in 2005.

However, a preliminary comparison has been carried out by CEN/TC 51 of the main features of the bulk diffusion tests that are, or could be, relevant to concrete and its constituents that are either already published or are currently under development. The extraction conditions employed by the different methods, and that may have relevance to the bulk diffusion extraction procedure likely to be developed by CEN/TC 351, are summarised in Table 1 below.

Table 1 — Test methods

Main feature of method	**Test method**			
	CEN CR 351XX [3] **(unpublished)**	**Draft procedure in support of German DIBt regulations**[4]	**Dutch NEN 7345** [5]	**CEN/TC 292 draft compliance test for leaching from monolithic waste (2003)** [6]
Scope	Natural environment	Natural environment (groundwater & soil)	Natural environment	Natural environment
Test pieces	100 mm concrete cubes made in accordance with EN 12390-2	100 mm concrete cubes	Minimum dimension 40 mm	Minimum dimension 40 mm
Moulding requirements	Thorough washing, no release oil.	Shale oil must not be used	Method is generic and does not deal with concrete specifically	May be prepared by moulding, cutting or coring
Reference concrete composition	Test samples are production concretes	Cement content 280 kg/m^3 w/c 0.60 (or highest anticipated for use) Aggregate to DIN 4226	Method is generic and does not deal with concrete specifically	Method is generic and does not deal with concrete specifically
Curing conditions	Demoulded at 24 h 6 days in sealed polythene bag at	Demoulded at 24 h Up to 56 days in sealed plastics at (20 ± 2) °C.	Method is generic and does not deal with concrete specifically	Not specified but age must be recorded Test pieces to be stored in sealed containers at

	(20 ± 2) °C Minimum of 21 days at 65 % RH	Storage up to 91 days may be agreed		(20 ± 5) °C.
Number of test pieces	1	1	Minimum of 2	1 (but several can be in the one tank)
Age of test piece at test	Greater than 91 days Weight at test to be within 1 % of demould weight	Normally 56 days	Method is generic and does not deal with concrete specifically	Not specified but should be adequate to enable structure to have stabilised
Test pieces rinsed before testing	No	No	No	No. Dust removed with compressed air.
Tank details	Unplasticized plastics, sealable	PE, PTFE or glass	Unplasticised plastics sealable	Glass or plastics. Must be capable of containing applied vacuum
Washing of equipment	1M HNO_3 then demineralised water	Not specified	Not specified	Not specified but blank required
Leachant	Demineralised water with pH between 4 and 7	Demineralised water	Demineralised water with pH of (4 ±) 0.1	Demineralised water with pH between 5 and 7,5
Leaching temperature	(20 ± 2) °C	(20 ± 3) °C	(20 ± 2) °C	(20 ± 5) °C
Stirring	No	Yes	No	Yes
Initial vacuum for saturation	No	No	No	Yes
Liquid volume : test piece surface ratio (lm^{-2})	(In range 67:1 to 100:1)	80:1	Not specified	6
Liquid volume : test piece volume ratio	>4 < 6	Not specified	>4 <6	Not specified
Leaching periods	6h 24h 78h 7d 14d	1d 2d 4d 9d 16d 24d	6h 1d 2,25d 4d 9d 16d 36d 64d	6h 1d 2d
Tests on leachate	pH, conductivity, inorganic analysis	pH, conductivity, inorganic analysis, TOC	Not specified	pH, conductivity (redox potential may be measured)

Storage of leachate	Minimise headspace, for most cations acidify with HNO_3	Not specified	Not specified	Not specified
Blank correction	No	No	No	Max. of 20 % contamination by any component No correction for blank values
Limiting criteria	No	Yes	No	No
Expression of results	Area related release (mg/m^2)	Area related release (mg/m^2) at 56 days Calculation of effective diffusion coefficient Prediction of substance concentrations in contact with groundwater	Area related release (mg/m^2)	Concentration in (mg/l) for the specified contact times Area-related release (mg/m^2) Leached quantity (mg/kg)

Assessment of results

The assessment of the results produced by the test methods in relation to the potential impact on soil, groundwater or surface water is a regulatory matter. Assessment of the release scenarios in combination with current transfer models applied at national level in some EU Member States allows the calculation of concentrations of relevant (regulated) dangerous substances to be predicted at, either:

any distance from the point of contact between the construction product and the soil or groundwater, or;

for any period of time during which the construction product could be in contact with soil or groundwater.

In the former case, where the distance from the construction product (point of compliance) has been fixed by the competent authorities at a national level, the calculated concentrations can be compared with the respective limit values. Alternatively, the transfer modelling can be used to derive limit values to be applied at the laboratory level for those substances which have regulatory limits established at the national level.

1 Scope

This document outlines three test methods. The first is designed to test the constituents of concrete, not designated as WT products, using reference concrete matrices (control mixes and test mixes) wherein the release of (regulated) dangerous substances from the constituent under test, into soil, groundwater or surface water, can be determined. The types of constituent which can be tested using this method are as follows:

a) factory-made cements;

b) aggregates;

c) additions type I;

d) additions type II;

e) admixtures;

f) polymer modifiers;

g) fibres.

The second method, in normative Annex A, is designed to test factory made concrete products, not designated as WT products, as either test pieces sawn or cored from pre-hardened monoliths or as standard-sized moulded test pieces formed from proxy samples of fresh wet material taken from concrete used in the production of factory made items.

The third method, in informative Annex B, is designed to test concretes sampled in the fresh wet state or pre-packaged state, not officially classified as WT products, as standard-sized moulded test pieces.

All three methods produce eluates that may be used for the purposes of characterisation testing, initial type testing (ITT) or further testing (FT) of either the constituents of concrete identified in this Scope or of production concretes.

2 Normative references

The following 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.

EN 197-1, *Cement – Part 1: Composition, specifications and conformity criteria for common cements*

EN 206-1:2000, *Concrete – Part 1: Specification, performance, production and conformity*

EN 480-1, *Admixtures for concrete, mortar and grout – Test methods – Part 1: Reference concrete and reference mortar for testing*

EN 1015-2, *Method of test for mortar for masonry – Part 2: Bulk sampling of mortars and preparation of test mortars*

EN 12350-1, *Testing fresh concrete – Part 1: Sampling*

EN 12350-2, *Testing fresh concrete – Part 2: Slump test*

EN 12350-6, *Testing fresh concrete – Part 6: Density*

EN 12390-1, *Testing hardened concrete – Part 1: Shape, dimensions and other requirements for specimens and moulds*

EN 12390-2, *Testing hardened concrete – Part 2: Making and curing specimens for strength tests*

EN 12390-7:2000, *Testing hardened concrete – Part 7: Density of hardened concrete*

EN 12620, *Aggregates for concrete*

EN 13639, *Determination of total organic carbon in limestone*

3 Terms and definitions

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

3.1
appropriate body
certification body, inspection body or test laboratory, as relevant to a particular requirement

3.2
cementitious product
factory made product containing a cementitious material supplied in the hardened state with a formed surface prior to its incorporation into the construction works

3.3
cementitious material
material that contains a hydraulic cement in sufficient proportion to act as the main binder by forming a hydrate structure which governs the performance of the material after hardening

3.4
fresh concrete
concrete that is fully mixed and still in a condition capable of being compacted by the chosen method

3.5
test
technical operation that consists of the determination of one or more characteristics of a given product

3.6
test procedure
specified technical method for performing a test

3.7
sample
one or more units, or a specified quantity, drawn from a batch or lot for inspection, e.g. at the factory or in a laboratory

3.8
reference concrete
generic concrete mix (see clause 7) made from reference cement (7.3), reference aggregates (7.2) and specified mixing water (7.4), proportioned to a reference cementitious content (7.5) and specified slump class (7.5), which is used to provide a (specific) control mix (see 3.10) and a standardised matrix from which to derive a (specific) text mix (see 3.11)

3.9
reference constituent
generic reference cement (7.3) and generic reference aggregates (7.2) used within a reference concrete

3.10
control mix
specific reference concrete mix made from a particular reference cement and particular reference aggregates used to form a blank (i.e. a control) in which the constituent under test is absent

3.11
test mix
specific reference concrete mix into which the constituent (new/unapproved) under test has been incorporated either by addition or substitution of a particular reference cement or particular reference aggregates, in the fresh wet state

3.12
test piece
either a standard sized hardened concrete specimen formed from a test mix, a control mix or a production concrete sampled in the formless (i.e. fresh wet) state which, after curing, provides contact surfaces representative of the intended service condition for concrete or, alternatively, an irregular sized concrete specimen sawn/cored from a production concrete sampled in the pre-hardened/precast state

3.13
test water
water used for testing purposes

3.14
eluate
test water which has been in contact with a test piece under specified conditions

3.15
blank water
test water which has been kept at the same specified conditions as eluate water but without contact with the test piece

3.16
mixing water
tap water

3.17
tap water
drinking water distributed by a public supplier

4 Principle

The constituent under test is incorporated in a test mix of concrete obtained by either substituting or adding the constituent to a reference concrete mix based on controlled quantities of cementitious material and slump class. The test mix is moulded in the form of a standard sized cube and cured under specified conditions prior to being brought into contact with a temperature controlled test water of controlled/uncontrolled pH, under static/dynamic conditions.

In general, except where an unapproved factory made cement is the constituent under test, a control mix is also prepared using the reference concrete. The control mix is moulded in the form of a standard sized cube, cured and subjected to the same extraction procedure as the test mix. A control mix is not required where an unapproved factory made cement is under test because the reference aggregates, meeting the requirements, of 7.2 have been specified to make a negligible contribution to release of substances.

Appropriate corrections are made to the determined contents of substances in the eluates obtained from most test mixes by either subtracting appropriate contributions from the control mix in which the constituent under test is absent, or where appropriate, by subtracting contributions from a procedural blank prior to calculation of concentrations for each of a number of contact periods with test water.

It is assumed that for final assessment of release, each constituent is either subject to constituent-specific limit values (expressed as either μg/l in the eluate and/or as mg/dm^2 of surface) for specified substances or that appropriate classes of performance exist.

5 Reagents

5.1 General requirements

The apparatus list from the horizontal bulk diffusion test method to be developed by CEN/TC 351 will be reproduced here.

Use only reagents of analytical quality unless otherwise stated.

6 Apparatus

6.1 General

The apparatus list from the horizontal bulk diffusion test method to be developed by CEN/TC 351 will be reproduced here.

6.2 Method-specific apparatus

6.2.1 Balance, capable of weighing to an accuracy of ± 1 g.

6.2.2 Laboratory oven(s), capable of being set at (105 ± 5) °C.

6.2.3 Concrete mixer, laboratory size, capable of mixing a homogeneous concrete from its constituents.

6.2.4 Moulds, shall be made from alkali-resistant material that will not interfere with the chemical analysis of eluates. A mould shall give a test piece with total surface area of (6,0 ± 0,6) dm^2.

Where steel moulds conforming to the requirements of EN 12390-1 are used, the joints shall not be coated with any wax, oil or grease to achieve water tightness. The use of release agents is not permitted.

NOTE Square 100 mm high-density polyethylene (HDPE) containers, cut down to the height of 100 mm, have been found to be satisfactory for this purpose.

6.2.5 Constant temperature/constant humidity cabinet, capable of maintaining temperature at (20 ± 2) °C and relative humidity at both (65 ± 5) % and greater than 90 %. At the higher relative humidity, an incubator containing a tray of water may be used to cure test pieces.

7 Reference concrete

7.1 General

The reference concrete, specified herein, provides both a control mix (3.10) and the concrete matrix for test mixes (3.11).

Concrete mix design, concrete mixing and testing shall only be carried out under the supervision of an experienced concrete technologist. Test mixes containing the maximum dose of some additions, admixtures and polymers may be very difficult to control under the specified test requirements and it is recommended that a concrete technologist with specific experience in these types of constituent is involved in such tests.

It is recommended that the laboratory should be a participant in a certified laboratory management scheme.

7.2 Reference aggregate

The reference aggregate, which may comprise a mixture of different coarse and fine fractions, in the control mix shall be a natural normal-weight aggregate conforming to EN 12620 with a water absorption less than 2 % by mass, a total organic carbon content (TOC) less than 0,1 % by mass and an aggregate grading that meets the requirements of EN 480-1. It shall be in an oven-dry condition achieved by drying at (105 ± 5) °C until constant mass is obtained.

The TOC content of the aggregate shall be determined in accordance with EN 13639. In the case of separate determinations for the coarse and fine fractions the TOC content of the aggregate (combined fractions) shall be calculated in accordance with the proportions by mass of the coarse and fine fractions in the aggregate.

7.3 Reference cement

The cement in the control mix shall meet the requirements of EN 197-1 for a CEM I cement.

7.4 Mixing water

Water, suitable for drinking, shall be used in the control and in the test concrete mixes.

7.5 Reference concrete mix proportions

The reference concrete control mix shall be proportioned to a cement content of (300 ± 20) kg/m^3 and slump of 50 mm to 90 mm, (slump class S2 in EN 206-1), determined in accordance with the method described in EN 12350-2.

NOTE Concrete placed in contact with either soil, groundwater or surface water will experience an environmental exposure that can be described as wet, rarely dry. This exposure condition is classified as XC 2 in EN 206-1, a classification that includes the following informative example: *Concrete surfaces subject to long-term water contact. Many foundations.* Consequently, a concrete conforming to the XC 2 classification in EN 206-1 is likely to be the minimum quality to be placed in contact with soil, groundwater or surface water. In addition, a recommended minimum CEM I cement content of 280 kg/m^3 is given in EN 206-1 for the XC 2 classification and this recommended minimum has been taken into account in setting the target CEM I cement content for use in the reference (control) mix, and most test mixes, at (300 ± 20) kg/m^3.

Prepare the control and test mixes (see Clause 9) observing the mix design principles in informative Annex C of this CEN TR.

8 Sampling of constituents

Sample constituents at the point of release of the factory or production facility in accordance with the relevant product standard, system standard or European or national regulations, whichever is appropriate.

Take care that the transport conditions do not influence the test results.

If it is necessary to store samples or test pieces before testing, ensure that they are protected from contamination taking into account any documented instructions that are provided.

Where appropriate, clean storage containers using the same procedures as are used for the test containers.

9 Control mix, test mixes and test pieces

9.1 General

Test pieces are of two types, those formed from the control mix and those formed from the test mix incorporating the constituent under test.

9.2 Test piece preparation

9.2.1 Test pieces made from control mix

Control test pieces are formed from (hardened and cured) control mixes using the reference constituents (Clause 7) in the proportions specified in 7.5. Under some circumstances, see 9.2.2.5 and 9.2.2.8, the use a particular constituent (e.g. some type II additions or some fibres) may require the use of a separate plasticizing or superplasticizing admixture (e.g. for efficient dispersal), in these cases the admixture is added to the control mix at the same dosage as that added to the test mix.

9.2.2 Test pieces made by incorporating an unapproved constituent in the reference concrete

9.2.2.1 General

Constituents to be assessed for approval are incorporated into the reference concrete either by substitution (wholly or in part) or by addition (as appropriate to the type of constituent) in order to produce a test mix which when hardened and cured forms a test piece.

Only one constituent may be added or substituted in any individual test mix.

In all cases, demoulding of test pieces should only be undertaken when the mix has hardened sufficiently so that demoulding can be carried out without causing damage to the surface of the test piece.

9.2.2.2 Test mix for factory-made cements

Replace, on a mass basis, all the reference CEM I cement in the reference concrete mix by the factory-made cement under test.

Maintain slump in the range 50 mm to 90 mm, determined in accordance with EN 12350-2, observing the mix design principles in informative Annex C of this CEN TR.

9.2.2.3 Test mix for aggregates

Replace some or all of the reference aggregate, on a volume basis, by the aggregate under test, prepared to meet the grading requirements of EN 480-1.

Maintain slump in the range 50 mm to 90 mm, determined in accordance with EN 12350-2, observing the mix design principles in informative Annex C of this CEN TR.

It may be necessary to make provision for the aggregate grading to be adjusted by the addition of fines which meet the physical and chemical requirements of clause 7.2. It is important that the test aggregate is prepared in such a way that the material used is representative of the bulk composition and that, for example, fines are not discarded.

9.2.2.4 Test mix for type I additions

Replace, on a mass basis, the fine fraction of the reference aggregate by the type I addition under test, at the maximum proportion (as a percentage of the total aggregate) specified for use of the type I addition.

Maintain slump in the range 50 mm to 90 mm, determined in accordance with EN 12350-2, observing the mix design principles in informative Annex C of this CEN TR.

9.2.2.5 Test mix for type II additions

Replace the reference cement with the maximum proportion of the type II addition calculated in accordance with the relevant k-value concept in 5.2.5 of EN 206-1:2000 used to establish suitability for use in concrete. Where the type II addition requires the use (e.g. for efficient dispersal) of a separate plasticizing or super plasticizing admixture, incorporate the admixture in both the control mix and the test mix using the minimum dosage required to achieve the target slump level.

NOTE In the case of type II additions and the use of 5.2.5 of EN 206-1:2000, the minimum content of cementitious material in a test mix, i.e. CEM I reference cement plus an equivalent quantity of type II addition, will vary from the target value set at 300 kg/m^3 for the control mix.

The introduction of a type II addition, with or without a plasticizing admixture, may result in extended setting times for test mixes.

9.2.2.6 Test mix for admixtures

Add the admixture under test to the reference concrete at the maximum dosage recommended by the manufacturer.

In general, maintain slump in the range 50 mm to 90 mm, determined in accordance with EN 12350-2, observing the mix design principles in informative Annex C of this CEN TR.

Where a high dosage of some admixtures, e.g. a superplasticising admixture, is recommended, it may be difficult to judge the water reduction needed due to thixotropic effects. In these cases, a slump in the range 50 mm to 150 mm is permitted for test mixes.

Where the maximum dosage recommended for some admixtures leads to retardation or other effects under the conditions for test piece preparation, modifications to the dose or test procedures may be agreed with the manufacturer and recorded in the test report.

Where the admixture results in rapid setting (for example admixtures for sprayed concrete) the concrete components may be conditioned at (5 ± 1) °C prior to mixing.

High dosages of admixtures can have retarding effects and may result in extended setting times for test mixes.

9.2.2.7 Test mix for polymer modifiers

Add the polymer modifier under test to the reference concrete at the maximum dosage recommended by the manufacturer.

In general, maintain slump in the range 50 mm to 90 mm, determined in accordance with EN 12350-2, observing the mix design principles in informative Annex C of this CEN TR.

For some dosages of polymer modifiers it may be difficult to maintain slump in the range specified. In these cases a slump in the range 50 mm to 150 mm is acceptable for test mixes.

9.2.2.8 Test mix for fibres

Add the fibres under test to the reference concrete at the maximum dosage recommended by the manufacturer. Where efficient addition of the fibres requires the use (e.g. for efficient dispersal) of a separate plasticizing or super plasticizing admixture, incorporate the admixture in both the control mix and the test mix using the minimum dosage required to achieve the target slump level.

In general, maintain slump in the range 50mm to 90mm, determined in accordance with EN 12350-2, observing the mix design principles in informative Annex C of this CEN TR.

For some dosages of fibres it may be difficult to maintain slump in the range specified. In these cases a slump in the range 25mm to 150mm is acceptable for test mixes.

10 Preparation of concrete test pieces

10.1 Moulds for test pieces

Moulds for preparing test pieces shall be made from alkali-resistant material that will not interfere with the chemical analysis of eluates. Moulds shall be cleaned before use. A mould shall give a test piece with total surface area of (6,0 ± 0,6) dm^2.

Where steel moulds conforming to the requirements of EN 12390-1 are used, the joints shall not be coated with any wax, oil or grease to achieve water tightness. The use of release agents is not permitted.

NOTE Square 100 mm high-density polyethylene (HDPE) containers, cut down to the height of 100 mm, have been found to be satisfactory for this use.

10.2 Concrete mixing and compacting procedure

An experienced concrete technologist shall design the concrete and supervise the mixing of the control and test mixes.

NOTE 1 An experienced concrete technologist is a person who either designs concrete mixes, or supervises the preparation of concrete mixes, in either a laboratory context or production facility, as part of their day-to-day professional activities.

It is recommended that the laboratory should be a participant in a certified laboratory management scheme.

The constituents shall be conditioned and mixed in accordance with EN 480-1 at a temperature of (20 ± 2) °C.

Where the slump level lies outside the range 50 mm to 90 mm, observe the mix design principles of informative Annex C of this CEN TR and prepare a new mix. In the case of certain admixtures, and when polymer modifiers or fibres are incorporated in the mix it is permitted to widen the acceptable slump range. See 9.2.2.6, 9.2.2.7 and 9.2.2.8.

Determine the fresh concrete density according to EN 12350-6 and recalculate the cementitious content in accordance with C.2.4. If the recalculated cementitious content lies outside the specified tolerance of ± 20 kg/m^3 in comparison with the target value (generally 300 kg/m^3 except in the case where type II additions are under test) discard the concrete mix and prepare a further batch making appropriate adjustments to the quantities of the constituents.

NOTE 2 Where a type II addition is incorporated in the mix the cementitious content is calculated on the basis of equivalent cement content (CEM I plus equivalent type II addition content).

If the cementitious content lies within the specified tolerance fill the mould(s) (number to be appropriate to the substance to be determined) with the mix and compact in accordance with EN 12390-2.

10.3 Curing and storing of test pieces

Cover the mould with a clean impermeable material which does not react with cement and transfer the freshly prepared test specimen to a constant temperature and constant humidity cabinet or an incubator containing a tray of water. Maintain the test specimen at a temperature of (20 ± 2) °C and relative humidity greater than 90 % for two days.

After curing for two days, demould the test piece and weigh to an accuracy of ± 1g and record the mass at demould. Ensure that the record of the mass at demould accompanies the test piece as it proceeds to the extraction procedure. After demould, place the weighed test piece in a clean food grade plastics bags, and seal. Store the test piece:

— a) either under environmental control at a temperature of (20 ± 2) °C and relative humidity of (65 ± 5) %,or;

— b) alternatively, place the sealed bag in another bag, and seal;

for a further period as specified in product standards, European or national regulations, as appropriate.

NOTE Storage conditions are designed to maintain test pieces in the same moisture condition prevailing at the end of curing. Although placing in a sealed bag should be adequate, additional precautions have been included in case of failure of the seal or imperfect closure.

11 Extraction procedure

11.1 General

Just prior to carrying out the extraction procedure remove the test piece from the sealed bag(s), weigh to an accuracy of ± 1 g and record the mass. Compare the mass with the mass recorded at demould (see 10.3). Where the mass lies within ± 1 % of the mass at demould, carry out the extraction procedure in 11.2. Where the mass lies outside ± 1 % of the mass at demould, reject the test piece.

11.2 Extraction

Adopt the extraction procedure in the horizontal bulk diffusion method to be developed by CEN/TC 351.

Subsequent to extraction, it is recommended that the eluate be filtered immediately after sampling in order to remove any particulates which might otherwise be dissolved during acidification of the eluate. Experience indicates a pore size of 0,22 micrometres is suitable.

11.3 Blank controls

11.3.1 Blank to be used with factory made cements

For each contact period, prepare blank water (3.15) by carrying out a blank test procedure using the same extraction conditions (test water, test temperature, contact periods used etc.) but omitting the test piece.

11.3.2 Blank to be used with aggregates, additions type I, additions type II, admixtures, polymer modifiers and fibres

Carry out a blank test on the test piece prepared from the control mix (see 9.2.1) using the same conditions as those for the test piece prepared from the test mix.

12 Assessment of unapproved constituents

12.1 General

The procedure (subtractive) for assessing unapproved constituents is based on the assumption that constituent-specific limit values or appropriate performance classes will be available for substances released from constituents where tested within a matrix/reference concrete mix that is broadly representative of intended use.

As a consequence of the micro-structural modifications that could occur in the bulk cementitious paste as well as in the bonding zone between paste and aggregate when concrete constituents are inter-changed, the mathematical subtraction of the data related to control and test mixes can give negative values for release of specified substances.

Any assessment of final results should make due allowance for the uncertainty of measurement of the method.

NOTE Uncertainty of measurement is the statistical criterion to be used where test results are to be compared with a regulatory or specification limit. An equation suitable for calculating uncertainty as a critical difference at the 95 % level of confidence is given in 4.2.3 of ISO 5725-6:1994 [1] for results for comparison with a reference value for one laboratory.

12.2 Assessment of factory made cements

Determine contents of specified substances released from the factory made cement by carrying out the extraction procedure on the test piece prepared from the test mix (9.2.2.2).

NOTE The reference aggregates make a negligible contribution to substances released from the test mix and therefore the results are cement-specific and can either be compared with any cement–specific limit values set for substances released or compared with any generic limit values set for substances released from concrete.

12.3 Assessment of aggregates, type I additions, type II additions, admixtures, polymer modifiers and fibres

Determine contents of specified substances released from these constituents by carrying out the extraction test procedure on the control mix (Clause 7) and the relevant test mix (i.e. 9.2.2.3, 9.2.2.4, 9.2.2.5, 9.2.2.6, 9.2.2.7 and 9.2.2.8). Subtract results obtained for control mixes from those obtained from test mixes.

13 Analysis

Carry out the required analysis on the eluates using the analytical methods specified in referring documents. Determine at the end of each extraction period the concentration of the substance being measured.

NOTE 1 General guidance on analytical performance requirements such as detection limit and accuracy is contained in Guide to analytical Quality Control for water analysis, ENV ISO 13530 [2].

NOTE 2 If eluates are not analysed immediately then ensure that the storage time and conditions do not adversely affect the analytical result.

14 Calculation of results

14.1 Calculation of the concentration of the substances in the eluate contributed by the unapproved constituent

NOTE 1 The release of substances into water depends on the type of material and the extraction conditions: temperature, contact time, S/V ratio and whether the water is static or flowing. For test conditions and constant temperature, the increase in the concentration of the substance in the test water is asymptotic. However, for practical purposes the increase with time is assumed to be linear.

Calculate for each eluate the concentration of the measured substance as follows:

$$c_n^T = a_n^T - b_n^T - d_n^T \quad (1)$$

where

c^T_n is the concentration of the measured substance in mg/l;

a^T_n is the concentration of the substance in mg/l measured in the eluate;

b^T_n is the concentration of the substance in mg/l measured in the blank water;

d^T_n is the concentration of the substance in mg/l measured in the control mix eluate.

NOTE 2 In the case where the unapproved constituent is a factory made cement, d^T_n is set to zero (see 11.3.1).

NOTE 3 In the case where the unapproved constituent is anything other than a factory made cement, b^T_n is not determined and is set to zero (see 11.3.2).

NOTE 4 In the case where the unapproved constituent is a type II addition, d^T_n is factored for the proportion of CEM I cement in the test mix.

For the conditions

T is the test temperature;

n is the sequence number of the extraction period.

14.2 Calculation of the rate of release of the measured substances

Calculate for each eluate the rate of release M^T_n for a substance from the concentration c^T_n as follows:

$$M_n^T = \frac{c_n^T}{(S/V \cdot t)} \qquad (2)$$

where

M^T_n is the rate of release for the n'th extraction period in $mg.dm^{-2}.day^{-1}$;

t is the duration of the extraction period in days at the defined temperature of extraction;

S/V is the ratio of the surface area of the test piece (i.e. 6,0 dm^2 in the case of a 100 mm cube) to the volume of test water, in dm^{-1}.

14.3 Calculation of the mean rate of release

Where extraction has been carried out in duplicate, calculate the arithmetic mean rate of release $\overline{M}^T_n$ for replicate values of M^T_n for each eluate.

15 Expression of results

The provisions for this clause will be reproduced from the horizontal bulk diffusion test method to be developed by CEN/TC 351.

16 Test report

The test report shall include the following information:

16.1 General information

a) date of issue;

b) name and address of testing laboratory and location where the test was carried out when different from the address of the testing laboratory;

c) unique identification of report (such as serial number) and of each page, and total number of pages;

d) name and address of client;

e) description and identification of the sample/test piece;

f) signature and title or equivalent marking of person(s) accepting technical responsibility for the test report and date of issue;

g) statement to the effect that the test results relate only to the test piece(s) tested;

h) statement that the report shall not be reproduced except in full without the written approval of the testing laboratory.

16.2 Information on the product

a) trade name or designation of the product or material;

b) complete identification and date of receipt of sample/test piece;

c) details of the test piece preparation;

d) mass of the test piece at demould and the mass of the test piece just prior to extraction;

e) name of the manufacturer for the product, the place of manufacture and date and, where relevant, the body submitting the sample and, where relevant, the body responsible for preparing the samples/test pieces;

f) description of the sampling procedure, where relevant.

16.3 Information on reference concrete mixes and test pieces

Where applicable, report any additional information on the reference concrete and test piece.

16.4 Information on the test procedure

a) reference to this document and to the referring product or system standard or European or national regulation as appropriate;

b) dates of start and completion of the test;

c) number of test pieces used together in the extraction procedure;

d) volume of the test water (V) in litres;

e) surface area of test piece exposed to the test water, S, in square decimetres calculated from the actual dimensions of the test pieces;

f) actual S/V ratio used in the procedure;

g) test waters and test temperature;

h) any deviation from the test procedure specified in this document;

i) any factors which may have affected the results, such as any incidents or any operating details not specified in this document.

16.5 Test results

a) number of tests carried out;

b) test results and calculated values reported in tabular form (see example below);

c) European Standard for the analytical method used to produce the test results;

d) general performance of the analytical method in the European Standard e.g. limit of detection, precision estimates, statement of uncertainty etc.

Table 2

	Sequence number of extraction period (n)			
	1	2	3	X[a]
a^T_n 1				
2^b				
b^I_n				
c^T_n 1				
2^b				
M^T_n 1				
2^b				
$\overline{M^T_n}$				

[a] additional extraction periods where specified

[b] duplicates where specified

17 Test performance (precision estimates and uncertainty)

Before the methods in this document can be adopted by CEN as European Standard methods, mandate M/366 requires the method to be validated in terms of variability and robustness, herein interpreted to mean that precision estimates for repeatability and reproducibility plus a statement of uncertainty are needed.

Annex A
(normative)
Testing for release of (regulated) dangerous substances from pre-hardened concrete products formed in the factory

A.1 Sampling, transport, test piece preparation and storage

A.1.1 Principle

Factory made pre-hardened concrete products are generally sampled in the hardened state as suitably sized test pieces of appropriate dimensions produced by coring/sawing but may also be sampled indirectly (proxy samples) in the fresh wet state and formed into standard sized cubes.

Test pieces that have been sampled in the pre-hardened state or that are formed as cubes are cured under conditions that are representative of those used in the manufacture of the factory-made item that they represent.

The extraction procedure is the 'horizontal bulk diffusion method' to be developed by CEN/TC 351 Construction products – Assessment of release of dangerous substances established in 2005. The concentrations of specified substances are determined in each eluate.

In order for test results to be assessed, it is assumed that either generic limit (e.g. threshold) values or appropriate classes of performance are available for release of specified substances from concrete.

A.1.2 Sampling

Carry out sampling of factory made pre-hardened concrete products at the point of release of the factory/plant in accordance with the relevant product standard or European or national regulations, as appropriate.

Where specified, factory made pre-hardened products may be sampled indirectly as proxy samples (fresh wet material) for preparation as test pieces.

A.1.3 Test piece preparation

A.1.3.1 Products sampled as formed in the factory

A.1.3.1.1 General

Test pieces shall be prepared in duplicate and be monolithic, undamaged and representative in structure, composition and homogeneity of the material from which they were prepared.

A.1.3.1.2 Test pieces

Test pieces may be prepared by coring or sawing sections from pre-hardened products using tap-water lubrication/cooling or may take the form of standard sized cubes where sampled indirectly (proxy samples) in the fresh wet state.

The minimum dimension of a test piece shall be 40 mm.

Test pieces shall be cured under conditions that are representative of those used in the manufacture of the factory-made item that they represent. Ensure that the minimum age of the test piece, at test, conforms to that recommended by the manufacturer for the product to be ready for use.

At the end of the curing period, test pieces shall be weighed to an accuracy of ± 1 g and the mass recorded. Ensure that the record of the mass accompanies the test piece as it proceeds to the extraction procedure. Place the weighed test piece in a clean food grade plastics bags, and seal. Store the test piece:

a) either under environmental control at a temperature of (20 ± 2) °C and relative humidity of (65 ± 5) %,or;

b) alternatively, place the sealed bag in another bag, and seal;

for a further period as specified in product standards, European or national regulations, as appropriate.

NOTE Storage conditions are designed to maintain test pieces in the same moisture condition prevailing at the end of curing. Although placing in a sealed bag should be adequate, additional precautions have been included in case of failure of the seal or imperfect closure.

Ensure that the surfaces of the test pieces intended to come into contact with the test water are free from any contamination e.g. adhesive tape, labels, ink or pencil marks.

The age at which a test piece is tested shall be reported.

A.1.3.1.3 Volume of a test piece

The volume (V_p), in litres, of a test piece shall be determined in accordance with 7.5 or 7.6 of EN 12390-7:2000.

The reference method (water displacement), given at 7.7 in EN 12390-7:2000 should not be used in order to minimize contact between water and a test specimen prior to extraction.

A.1.3.2 Materials sampled in the fresh wet state

A.1.3.2.1 General

Sample fresh wet concrete in accordance with EN 12350-1.

Where appropriate, clean storage containers using the same procedures that are used for cleaning the test containers.

A.1.3.2.2 Moulds for test pieces

Moulds for preparing test pieces shall be made from alkali-resistant material that will not interfere with the chemical analysis of eluates. A mould shall give a test piece with total surface area of (6,0 ± 0,6) dm^2.

Where steel moulds conforming to the requirements of EN 12390-1 are used, the joints shall not be coated with any wax, oil or grease to achieve water tightness. The use of release agents is not permitted.

NOTE Square 100 mm high-density polyethylene (HDPE) containers, cut down to the height of 100 mm, have been found to be satisfactory for this purpose.

A.1.3.2.3 Compaction procedure

Fill the mould(s) (number to be appropriate to the substance to be determined) with the mix and compact in accordance with EN 12390-2. Cure test pieces in accordance with A.1.3.4.

A.1.3.3 Curing of moulded test pieces

The test pieces shall be cured under conditions that are representative of those used in the manufacture of the factory-made item that they represent.

A.1.3.4 Packaging and transport of test pieces

Where testing requires transfer of test pieces from the place of making to a test laboratory, test pieces shall be maintained in sealed food grade plastics bags and packaged securely in order to minimise damage in transit.

A.2 Extraction

Carry out the extraction procedure in accordance with Clause 11.

For each contact period, carry out a blank test procedure using the same extraction conditions (test water, test temperature, contact periods used etc.) but omitting the test piece.

A.3 Analysis

Carry out the required analyses of the eluates in accordance with Clause 13.

A.4 Calculation of test results

Calculate test results in accordance with Clause 14.

A.5 Test report

Prepare test reports in accordance with Clause 16.

A.6 Test performance (precision estimates and uncertainty)

Express test performance in accordance with Clause 17.

Annex B
(informative)
Testing for release of (regulated) dangerous substances from hardened test pieces representative of fresh wet concretes or pre-packaged concretes

B.1 Sampling, transport, test piece preparation and storage

B.1.1 Principle

Production concretes (i.e. concretes as they are placed on the market) are supplied in three forms; as pre-hardened products formed in the factory (see normative Annex A); as fresh wet materials or as pre-packaged products.

For release of substances, materials such as fresh wet concretes supplied in a formless state are sampled in the fresh wet condition and formed into standard sized cubes. Whereas, pre-packaged products are sampled as whole bags of product, gauged with an appropriate quantity of water, mixed to form a fresh wet mix and formed into standard sized cubes.

Test pieces are cured under specified conditions, either at a production facility (e.g. fresh wet ready-mixed concrete), on site or as otherwise agreed (e.g. fresh wet site-mixed concrete), or in a laboratory, as appropriate to the type of product, prior to being brought into contact with a temperature controlled test water of controlled/uncontrolled pH under static/dynamic conditions in the extraction procedure.

The extraction procedure is the 'horizontal bulk diffusion method' to be developed by CEN/TC 351 *Construction products – Assessment of release of dangerous substances*, established in 2005. The concentrations of specified substances are determined in each eluate.

In order for test results to be assessed, it is assumed that either generic limit (e.g. threshold) values or appropriate classes of performance are available for release of specified substances from concrete.

B.1.2 Sampling

Carry out sampling of concrete, whether in the fresh wet state or pre-packaged, at the point of release of the factory/plant in accordance with the relevant product standard or European or national regulations, as appropriate.

B.1.3 Test piece preparation

B.1.3.1 General

Test pieces should be prepared in duplicate and be monolithic, undamaged and representative in structure, composition and homogeneity of the material from which they were prepared.

B.1.3.2 Materials sampled in the fresh wet state

B.1.3.2.1 General

Sample fresh wet concrete in accordance with EN 12350-1. Trial mixes representative of production concretes are permitted.

Where appropriate, clean storage containers using the same procedures that are used for cleaning the test containers.

B.1.3.2.2 Moulds for test pieces

Moulds for preparing test pieces should be made from alkali-resistant material that will not interfere with the chemical analysis of eluates. A mould should give a test piece with total surface area of (6,0 ± 0,6) dm^2.

Where steel moulds conforming to the requirements of EN 12390-1 are used, the joints should not be coated with any wax, oil or grease to achieve water tightness. The use of release agents is not permitted.

NOTE Square 100 mm high-density polyethylene (HDPE) containers, cut down to the height of 100 mm, have been found to be satisfactory for this purpose.

B.1.3.2.3 Compaction procedure

Fill the mould(s) (number to be appropriate to the substance to be determined) with the mix and compact in accordance with EN 12390-2. Cure test pieces in accordance with B.1.3.4

B.1.3.3 Products sampled pre-packaged in bags

B.1.3.3.1 General

Pre-packaged concretes include dry single pack concrete products and double packed products in which the aggregate is moist and the cement is contained within an impermeable inner bag.

Sample pre-packaged concretes in accordance with EN 1015-2.

Where appropriate, clean storage containers using the same procedures that are used for cleaning the test containers.

B.1.3.3.2 Moulds for test pieces

Mould should be specified in accordance with B.1.3.2.2.

B.1.3.3.3 Gauging with water and mixing procedure

Gauge the product with a quantity of water as advised in manufacturer's instructions and carry out mixing operations in accordance with the procedure in EN 1015-2,

Where no instructions are provided, gauge the product with a quantity of water, which after carrying out mixing operations in accordance with the procedure in EN 1015-2, gives a slump of 50 mm to 90 mm (slump class S2 in EN 206-1) determined in accordance with the method described in EN 12350-2.

B.1.3.3.4 Compaction procedure

Fill the mould(s) (number to be appropriate to the substance to be determined) with the mix and compact in accordance with EN 12390-2.

B.1.3.4 Curing of moulded test pieces

Cover the mould with a clean impermeable material which does not react with cement and transfer the freshly prepared test pieces to a constant temperature and constant humidity cabinet or an incubator containing a tray of water. Maintain the test pieces at a temperature of (20 ± 2) °C and relative humidity greater than 90 % for two days.

The maximum permissible delay between sampling of the fresh product and the transfer of the filled moulds to the controlled temperature and humidity environment is four hours.

After curing for 2 days, demould the test piece and weigh to an accuracy of ± 1g and record the mass at demould. Ensure that the record of the mass at demould accompanies the test piece as it proceeds to the

extraction procedure. After demould, place the weighed test piece in a clean food grade plastics bags, and seal. Store the test piece:

a) either under environmental control at a temperature of (20 ± 2) °C and relative humidity of (65 ± 5) %,or;

b) alternatively, place the sealed bag in another bag, and seal;

for a further period as specified in product standards, European or national regulations, as appropriate.

NOTE Storage conditions are designed to maintain test pieces in the same moisture condition prevailing at the end of curing. Although placing in a sealed bag should be adequate, additional precautions have been included in case of failure of the seal or imperfect closure.

B.1.3.5 Packaging and transport of test pieces

Where testing requires transfer of test pieces from the place of making to a test laboratory, test pieces shall be maintained in sealed food grade plastics bags and packaged securely in order to minimise damage in transit.

B.2 Extraction

Carry out the extraction procedure in accordance with Clause 11.

For each contact period, carry out a blank test procedure using the same extraction conditions (test water, test temperature, contact periods used etc.) but omitting the test piece.

B.3 Analysis

Carry out the required analyses of the eluates in accordance with Clause 13.

B.4 Calculation of test results

Calculate test results in accordance with Clause 14.

B.5 Test report

Prepare test reports in accordance with Clause 16.

B.6 Test performance (precision estimates and uncertainty)

Express test performance in accordance with Clause 17.

Annex C
(informative)
Principles of laboratory concrete mix design to be applied for the assessment of new/unapproved constituents of concrete

C.1 Introduction

The design of concrete mixes is generally carried out in order to optimise the performance of the concrete in the fresh and hardened states. The concrete mixes required for the test procedure for assessing release of substances from new/unapproved constituents of concrete described in this document do not have to meet any prescribed performance in the hardened state, but have to be designed to permit the use of a controlled quantity of cementitious material and are subject to a requirement for consistence. In order to meet both requirements, it is essential that the content of the constituents can be determined accurately and that the concrete is correctly proportioned and mixed i.e. there should be no evidence of bleeding, lack of cohesion, tendency to segregate, excessive viscosity, etc.

Optimal proportioning of the mix is outside of the scope of this annex but some general principles of mix design are outlined in order to proportion the concrete efficiently and to improve the precision of the test method.

C.2 General methodology

C.2.1 General

The design of mixes should be carried out in the following manner:

a) calculate the proportions of the constituents on a theoretical basis;

b) prepare a trial mix and assess it;

c) check the proportioning;

d) correct the proportioning of the trial mix if necessary.

C.2.2 Theoretical calculation of the proportioning of the constituents

The theoretical calculation is carried out in order to determine the mass of the constituents needed to produce a specified volume of concrete. It is usual to assume a volume of 1 m³ as the reference.

Theoretically, the mass of the constituents (M_i) satisfies the following relationship:

Error! Objects cannot be created from editing field codes. [m³]
(C.1)

where

N = number of constituents;

ρ_i = absolute density of each constituent, in kg/m³;

NOTE 1 The density of each constituent can be measured or can be declared by the supplier of the constituent itself.

M_i = mass of each constituent, in kg;

NOTE 2 The amount of each constituent is defined from a prescribed limit (i.e. the cementitious content) or on the basis of laboratory experience in order to fulfil a defined performance (i.e. consistence). In general, the number of trial mixes needed to derive the final mix depends on the accuracy of this first estimation of quantities of constituents.

α = air (entrapped + entrained), in % by volume;

NOTE 3 The air that is entrapped[1] or intentionally entrained in the concrete is estimated for the first calculation. After the preparation of the trial mix the estimate can be checked by a direct measurement of the air content in accordance with EN 12350-7 [6].

Equation (C.1) is valid when the aggregate is dry. In the case of moist aggregate the masses of aggregate and water are modified in order to take into account the amount of water contained in the aggregate.

C.2.3 Trial mix and its assessment

The first trial mix is cast and assessed after determining the quantities of each constituent. If the trial mix is free from excessive bleeding, lack of cohesion or tendency to segregate etc, then the slump level and the density in the fresh state (D_f) are determined. The trial mix is then regarded as satisfactory if the slump falls within the specified limits and if the proportion of cementitious material, checked on the basis of the density in the fresh state, satisfies the specified tolerance. If one or more of these conditions is not met, then some adjustments are performed. Figure C.1 outlines a scheme that can be adopted for the assessment of the concrete and for the modification of its composition.

[1] A typical value for the entrapped air for common concretes is in the range 1 % to2 %.

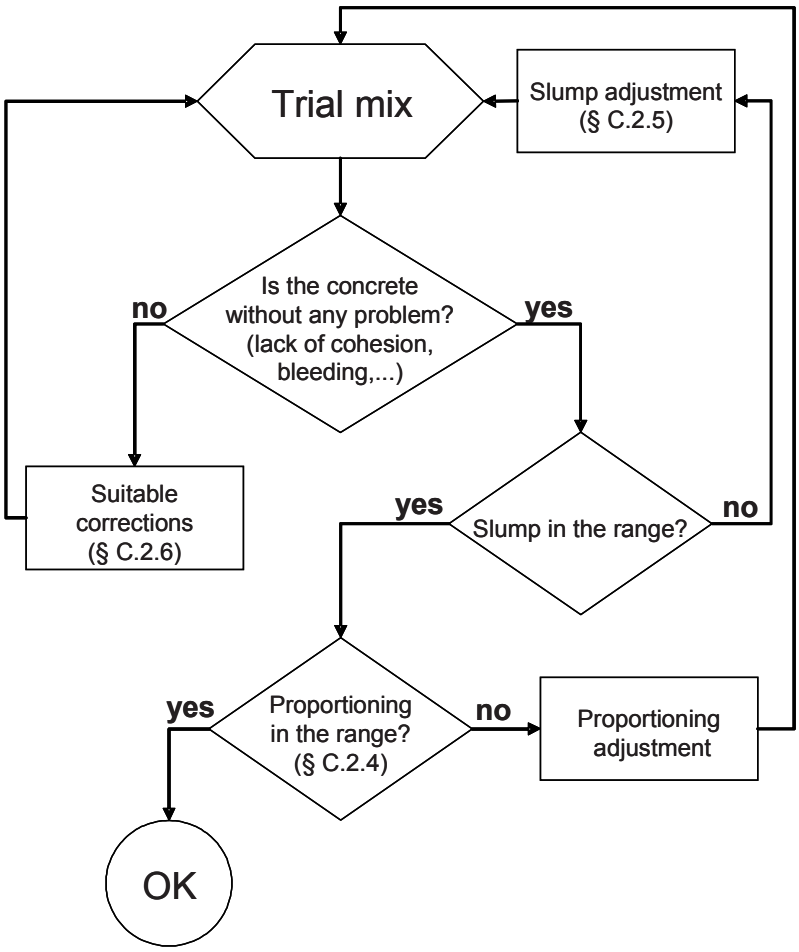

Figure C.1 - Scheme for modification of concrete composition in the assessment of constituents

C.2.4 Checking the proportioning

The check of the mix proportions, in particular the cementitious content, is carried out starting from the values of M_i and from the measured value of the density in the fresh state D_f.

The content C_i of each constituent, expressed in kg/m³, can be calculated from the following relationship:

Error! Objects cannot be created from editing field codes. [kg/m³]
(C.2)

where

Error! Objects cannot be created from editing field codes. [kg]
(C.3)

D_f = density of concrete in the fresh state measured according to EN 12350-6, in kg/m^3.

C.2.5 Adjustment of slump level

The adjustment of the slump level of the test concrete is carried out mainly by varying the water content. In practice many parameters influence the consistence of concrete, but in the context of this annex the most effective parameter that can be adjusted is the water content. Where an increased slump is required, more water is weighed into the mix. Conversely, if a reduction in slump is required, all the constituents of the mix in the relative proportions already determined, with the exception of water are weighed into the batch. In this way the volume of the batch and as a consequence the water content (expressed in terms of kg/m³) decreases. After achieving the desired level of slump, a further trial mix is prepared and, the resulting composition checked as described before.

In addition to, or as an alternative to, varying the water content of the mix, it is also possible to modify slump by varying the following parameters:

a) the cementitious content, within the permitted tolerance;

b) the grading curve of the aggregates, within the permitted range;

c) the type of aggregate. Rounded aggregates increase slump in comparison with crushed aggregates. In addition, the more porous aggregates lower slump value in comparison with more dense aggregates;

d) the fineness of cement. Finer cements tend to lower slump, whereas coarser cements tend to increase it.

Nevertheless these variations to the mix should only be seen as intermediate refinements to the starting composition. After achieving the desired level of slump, a further trial mix is prepared and, the resulting composition checked as described before.

NOTE The adoption of the mix proportioning determined with iterative additions of the constituents to the mixer, without a final check following the usual sequence of introduction of the constituents, is a cause of poor repeatability.

C.2.6 Adjustments to fresh wet properties other than slump

C.2.6.1 General

If the trial mix exhibits excessive bleeding, lack of cohesion, tendency to segregate or excessive viscosity the mix parameters can be varied in order to minimise these effects.

C.2.6.2 Bleeding/tendency to segregate

Bleeding describes the movement of water from within the mix towards the upper surface of test pieces after casting and is in general associated with segregation of concrete. The causes are usually failure to optimise the concrete proportioning or use of excessive vibration during the preparation of test pieces.

Possible actions to avoid this problem are:

a) increase the cementitious content within the permitted tolerance and/or use a finer cement;

b) modify the grading of the coarse aggregate within the permitted range and/or use an aggregate having a lower density;

c) increase the content of the fine aggregate within the permitted range;

d) decrease water content in order to reduce consistence to the lower permitted values.

C.2.6.3 Excessive viscosity

Where the concrete exhibits excessive viscosity the surfaces of cast test pieces may be inadequately finished. The causes are failure to optimise concrete proportioning, effects of the physical nature of some constituents or to prolonged mixing. Prolonged mixing can lead to grinding of constituents and an increase in fines content. In order to minimise any tendency for the trial mix to exhibit excessive viscosity, the following parameters can be varied:

a) reduce the cementitious content within the permitted tolerance and/or use a coarser cement;

b) change the grading of the coarse aggregate/sand within the permitted ranges to reduce the fines content or use an aggregate with a higher resistance to grinding/abrasion;

c) optimise the mixing operations in order to limit the mixing time.

C.2.6.4 Lack of cohesion

If the amount of paste (i.e. water plus cement plus any addition plus fines) is insufficient to fill the voids in the aggregates, then the concrete may exhibit poor cohesion. If this occurs, it is difficult to achieve full compaction of the test pieces and a good surface finish. Possible actions to resolve this problem are:

a) increase the cement and/or any addition content in the allowed range;

b) modify the grading of the coarse aggregate in the permitted range;

c) increase the content of sand in the permitted range.

C.2.7 Effects on mix design of constituents other than cementitious materials, aggregates and mixing water

Constituents other than cementitious materials aggregates and mixing water can have a significant impact on the properties of concrete in the fresh and hardened states. However, the possible effects on mix design of the inclusion of admixtures, polymer modifiers and fibres have not, been described in this annex because where new or unapproved constituents of these types are to be tested they are added at dosages recommended by the manufacturer. Addition of constituents at recommended dosages will restrict any variation in the content of such constituents and any adjustments to mix design will have to be made on the same bases as described in this annex.

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