

ICS 91.100.30

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

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Foreword

This document (CEN/TR 15678:2008) has been prepared by Technical Committee CEN/TC 51 “Cement and building 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 (l m⁻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) test mix (see 3.11)

3.9

reference constituent

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