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Harmonisation of test methods for the execution of the EU Construction Products Directive

Validation of a European leaching test for construction

Summery

by

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1 Introduction

Construction products in terms of the European Construction Products Directive (CPD) are building materials, components and constructions, which are produced for incorporation in a permanent manner in buildings and civil engineering works, as well as prefabricated constructions which are produced with building materials and components to be connected with the ground, for example prefabs, prefabricated garages and silos. A construction product may be placed on the market only if the conformity with the essential requirements is proved. Essential requirements in terms of the CPD are:

- No 1: Mechanical resistance and stability
- No 2: Safety in case of fire
- No 3: Hygiene, health and the environment
- No 4: Safety in use
- No 5: Protection against noise
- No 6: Energy economy and heat retention

Apart from the requirements traditionally defined in the building legislations the CPD explicitly demands construction works to be built with construction products that meet the hygiene, health and environmental requirements in force in their place of use and not to endanger the health of the inhabitants and the residents or the immediate environment of the construction work. Technical specifications (standards and technical approvals) which are harmonised according to the European Construction Products Directive must therefore include the requirements for the protection of the immediate surrounding area.

So far only few national regulations for construction products focus on the release of dangerous substances. Convenient European test methods to verify compliance with these regulations are missing. Therefore the harmonised European product standards adopted so far only make general references to existing national requirements. For the future compilation and revision of harmonised technical specifications the European Commission now demands to work out the corresponding verification concepts and associated verification instruments, such as methods of testing and analysis. To that end, the Commission issued at the beginning of the year 2005 the mandate M/366 "Development of horizontal standardised assessment methods for harmonised approaches relating to dangerous substances under the Construction Products Directive (CPD) – Emission to indoor air, soil, surface water and ground water". The mandate intends that the European Committee for Standardisation (CEN) is going to elaborate horizontal test and assessment methods. The concrete requirements regarding the environmental compatibility of construction products will continue to be defined nationally by the individual Member States.

To allocate the necessary generic, horizontal test and assessment methods for the implementation of mandate M/366 CEN set up the technical committee CEN/TC 351 "Construction products: Assessment of release of dangerous substances" in 2006. CEN/TC 351 shall bring together existing, diverging national test methods or European methods from other, linked fields of law, to harmonised test methods.

Three drafts for harmonised test methods are the main result of the work of CEN/TC 351 of the past few years. Two of these test methods are designed for the determination of the leaching of inorganic and organic substances from construction products with regard to soil and water. The methods of these test standards are a tank test "Generic horizontal dynamic surface leaching test (DSLT) for determination of surface dependent release of substances from monolithic or plate-like or sheet-like construction products" and a column test

"Generic horizontal up-flow percolation test for determination of the release of substances from granular construction products". The third test method is dealing with the determination of the release of volatile organic compounds into indoor air by means of test chamber measurements "Determination of emissions into indoor air".

Before the test methods worked out by CEN/TC 351 can get the status of a European standard, robustness tests and as a second validation step round robin tests are necessary. The aim of the robustness tests is to determine through variation of selected test parameters, for example the temperature of the water in the tank and column test or the air change rate in the test chamber, how sensitive the test method reacts to changes of the test conditions. After robustness testing the draft standards, where appropriate, will be adapted and submitted for publication as Technical Specification (TS).

As the funding of the European validation tests was not clear in the year 2009 the German Federal Environmental Agency granted this research project to support this process. The aim of the research project was to execute the validation tests for the tank test for selected monolithic construction products covered by the CPD. The full robustness tests were carried out for concretes manufactured of concrete constituents standardised under the CPD. Additionally a mineral mortar with a high amount of organic constituents was chosen to assess the suitability of the tank test for the investigation of the release of organic substances (TOC-release).

2 Execution of the research project and scientific-technical results

2.1 Concrete

2.1.1 Concrete production

For the investigations concerning the robustness of the tank test for concretes two different concretes were produced at three concretings. The concrete production and storage have been carried out according to part II of the principles for "Assessment of the effects of construction products on soil and groundwater" of the German Institute of Structural Engineering (DIBt). For the concrete production a Portland cement CEM I 42.5 R and a slag cement CEM III/A 42.5 N, which was produced with the same clinker as the Portland cement, were used. The properties specified in the standard, the chemical composition and the trace element content of the cements were determined as well as the consistency, the air content and the 28 d-, 56 d- and 91 d-compressive strength of the concretes.

2.1.2 Leaching results for the concretes

At the first concreting 28 concrete cubes with an edge length of 100 mm were produced with the Portland cement (cement content 280 kg/m³, water cement ratio w/c = 0.60) for the leaching tests. With eight test specimen the tank test was carried out according to the draft standard "Generic horizontal dynamic surface leaching test (DSLT) for determination of surface dependant release of substances from construction products". The age of the test specimen at the beginning of the leaching test was 56 days. The obtained leachates were investigated according to the DIBt-principles for inorganic parameters for which an insignificance threshold has been defined (see Table 2). Aside this, the sodium and potassium concentration in the leachates was determined. At the leaching test with the further 20 cubes of this concreting the test conditions were varied according to **Table 1**.

With the second concreting the influence of a repeated concrete production on the test results should be checked. For this three test specimen were produced with the Portland cement in the exact same manner as for the first concreting and the leaching tests were done in triplicate according to the draft standard (DSLT). With the concrete of the third concreting the influence of the cement type on the leaching behaviour should be investigated. For this six test specimen were produced with the slag cement. The leaching tests were done as double test according to the draft standard (DSLT) as well as at 15 °C and according to the Dutch standard NEN 7375, because these test variants have shown the greatest influence on the test results for the concretes of the first and second concreting.

Figure 1 shows exemplarily the accumulated leached amounts for chromium and **Figure 2** for sodium for all concretes of the first and second concreting. The interpretation of the test results has shown that the accumulated leached amounts at the eightfold test for the first concreting match well for the elements potassium, sodium, selenium and vanadium. For the other parameters a greater scattering is observed in contrast. This is particularly important if the individual test result is close to the quantification limit. The leaching tests done in triplicate for the second concreting generally show a better conformity as for the eightfold test of the first concreting. However, the leaching results show in many cases a greater difference between the first and the second concreting. In **Table 2** the relative standard deviations (coefficient of variation V) for the eightfold test of the first concreting, the triple test of the second concreting as well as for the combination of the first and second concreting are summarised.

Variation No.	Specification	
1	Decrease of the curing time to 28 days	
2	Increase of the curing time to 91 days	
3	Decrease of the testing temperature to 15 °C	
4	Increase of the testing temperature to 25 °C	
5	Duration of the specific leaching step according to the DAfStb-long term leaching test	
6	Duration of the specific leaching step according to the Dutch standard NEN 7375	
7	Decrease of the surface to volume ratio to 40 L/m ²	
8	Increase of the surface to volume ratio to 120 L/m ²	
9	Decrease of the pH value of the starting leachant to 4	
10	Increase of the pH value of the starting leachant to 10	

Table 1:	Variation of the test conditions ¹⁾ for the concrete tests	

¹⁾ all variations were done as double test

Whether the greater scatterings are due to the leaching test, the chemical analysis or to inhomogeneities of the concrete test specimen, could not be resolved in this research project.



Figure 1: Chromium release for all concretes of the first and second concreting



Figure 2: Sodium release for all concretes of the first and second concreting

Table 2:Relative standard deviation (coefficient of variation V) for the eight-
fold test of the first concreting, the triple test of the second concre-
ting as well as for the combination of the first and second concreting

Parameter	First concreting	Second concreting	First and second	
			concreting	
	Relative standard deviation (coefficient of variation V)			
	in %			
Barium (Ba)	36,3	8,98	139	
Lead (Pb)	19,8	13,5	56,3	
Chloride (Cl ⁻)	89,6	146	105	
Chromium (Cr)	17,2	3,88	55,2	
Potassium (K)	6,86	26,8	14,3	
Copper (Cu)	56,6	1,73	61,9	
Sodium (Na)	14,4	3,83	12,9	
Selenium (Se)	7,16	_1)	37,2	
Sulphate (SO ₄ ²⁻)	33,8	22,8	60,3	
Vanadium (V)	6,55	12,4	51,7	
Zinc (Zn)	34,9	3,95	28,8	

¹⁾ All values below the quantification limit

The leaching results for the concretes of the third concreting with the slag cement CEM III/A 42.5 N show a similar behaviour as the results for the concretes of the first and second concreting. The accumulated leached amounts for the parameters investigated are in the same order of magnitude as for the concretes with the Portland cement CEM I 42.5 R.

2.2 Mortar

2.2.1 Mortar production

For the investigations concerning the robustness of the tank test for mortars a reinforcing render (according to DIN EN 998), with a high amount of an organic additive, was used. The mortar production was carried out according to the manufacturer's instructions. The water/solid ratio was 1 to 5.3. The chemical composition and the trace element content of the reinforcing render was determined as well as the compressive and the bending tensile strength.

2.2.2 Leaching results for the mortar

From the prefabricated reinforcing render 16 mortar cubes with an edge length of 100 mm were produced for the leaching tests. With two test specimen the tank test was carried out according to the draft standard (DSLT). The age of the test specimen at the beginning of the leaching test was 56 days. In the obtained leachates the same parameters were investigated as for the tested concretes, in addition the TOC-release was determined.



Abbildung 3: TOC-release of the reinforced render for the variation of the test conditions according to Table1

At the leaching tests with the further 14 mortar cubes the test conditions were varied according to the positions No. 1, 3, 4, 5, 6, 7 and 8 of Table 1. **Figure 3** shows the cumulative TOC-release for all mortar tests for example.

3 Summary

The cumulative release for the investigated parameters have the same order of magnitude for the concretes and the reinforcing render. Thereby the results of the leaching tests show a very good conformity for potassium and sodium, whereas for the trace elements and anions a lager scattering was observed. This is particularly important if the individual test result is close to the quantification limit of the analysis method (ICP-OES). For the TOC-release of the reinforced render a very good conformity of the test results for the variation of the test conditions was observed, too (Figure 3). From this it can be deduced that the tank test (DSLT) is also appropriate for the investigation of the release of organic substances from construction products.

The results for the leaching of potassium, sodium and TOC show that the leaching test as such is robust and that for the conducted variation of the test conditions for these parameters only a minor scattering is observed. A further essential result of the investigations is that the releases for the elements potassium and sodium as well as for TOC, which where obtained by the Dutch standard NEN 7375 and the DAfStb-long term leaching test, are located in all cases approximately in the middle of the scattering for the realised variations of test conditions. This shows that the transmission of the results observed by the Dutch standard NEN 7375 and the DAfStb-long term leaching test on the 36-days-tank test according to the draft European standard (DSLT) should be possible relative easily.

From the presented results of the investigations it can be concluded that an amendment of the draft European standard (DSLT) with regard to the range of test conditions is not necessary. This result is a substantial contribution to the validation process.