Dutch Standard

NEN 7347

(en)

Uitloogkarakteristieken van vaste grond- en steenachtige bouwmaterialen en afvalstoffen - Uitloogproeven - Bepaling van de uitloging van anorganische componenten uit gecompacteerde korrelvormige materialen

Leaching characteristics of solids earthy and stony building and waste materials - Leaching tests - Determination of the leaching of inorganic components from compacted granular materials

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Table of Contents

Foreword

To determine the various aspects of the leaching behaviour (the leaching characteristics) of solid earthy and stony building and waste materials a series of steps shall be followed, in particular sampling, sample pretreatment, leaching tests, digestion and chemical analysis of the solid substance or the eluates. For each step one umbrella standard is/will be drawn up, in which general instructions are given. In this the relationship is given between all the standards falling under this step, each with a specific scope. To determine the leaching characteristics, the general instructions or the specific standards to which reference is made shall be followed with good consistency.

The general instructions for the choice and the applicability of the leaching tests are described in NEN 7340 and NEN 7370. Among other things these standards indicate the type of materials or components for which the various leaching tests can be used.

This standard describes a test that can be used to determine the leaching behaviour of granular materials under conditions where the leaching is mainly diffusion controlled. This standard, hereinafter called 'granular diffusion test', is based on the same principles and procedure as described in the diffusion test for moulded and monolithic waste materials according to NEN 7345 and NEN 7375. The granular diffusion test among other things provides the result of the cumulative emission from the effective area exposed to leaching of a mass of compacted granular material (in mg/m^2). Based on the results of the granular diffusion test a judgment can be formed of the time dependency of the leaching of a compacted granular material under practical conditions, where this leaching is a result of diffusion, in conditions in which diffusion is the determinant factor [1], [4].

The standards that characterise the various aspects of the leaching behaviour are produced and published in phases. This means that upon the publication of this standard, reference is not yet made in NEN 7370 and NEN 7340 to the present granular diffusion test and also that some of the relevant standards have not yet been published as a draft standard or standard, including the revision of NEN 7340. Users of this standard will for the missing aspects have to make their own choice of the methods to be used. Furthermore, standards and other publications that have been published in this respect and which are included in the bibliography can also be used.

The numbered sections are normative with the exception of the passages marked with the heading 'NOTE'; the annexes are informative.

Leaching characteristics of solid earthy and stony building and waste materials – Leaching tests – Determination of the leaching of inorganic components from compacted granular materials

1 Scope

This standard describes a laboratory test to determine the leaching of inorganic components from granular building and waste materials under diffusion controlled conditions.

For a specification of the materials with which experience has been acquired with the execution of the test according to this standard, as well as the conditions under which the test can be used, see Annex A and [1].

NOTE To determine the leaching of inorganic components from granular materials under percolating conditions, the column test according to NEN 7373 or according to NEN 7383 and/or the cascade test according to NEN-EN 12457 are referred to. To determine the leaching of inorganic components from moulded and monolithic waste materials NEN 7375 is referred to.

2 Normative references

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

NEN 1047:1967 *Instructions for statistical treatment of series of observations*

NEN 6411:1981 *Water – Determination of pH*

NEN 7360:1997 *Leaching characteristics of solid earthy and stony building and waste materials – Terms and definitions*

NEN-ISO 7888:1994 *Water – Determination of electrical conductivity*

NEN-EN-ISO 5667-3:2004 *Water quality – Sampling – Part 3: Guidance on the preservation and handling of water samples*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in NEN 7360 apply.

4 Principle

The purpose of the test is to simulate the diffusion controlled leaching of inorganic components from granular building and waste materials in an aerobic environment, as a function of time over a period of 64 days.

In the test the diffusion controlled leaching of a granular material is simulated by compacting a quantity of it in a cylindrical vessel and then exposing it on one side to a leaching fluid (pH-neutral; demineralized water) and refreshing the eluates at set times. The concentrations of the leached components in the successive eluate fractions obtained are measured. The pH value under which the leaching is carried out is imposed by the material itself. Based on the results of the test the leached quantity per unit area of each component analysed is calculated both per fraction and cumulatively. Parameters can be deduced from the course of the emission of components over time, including the degree of surface wash-off and the effective diffusion coefficient, with which leaching can also be estimated in the longer term.

NOTE In the past the diffusion test according to NVN 7347 had to be carried out with demineralized water which was acidified with nitric acid to pH = 4 ± 0,1. In [11] the change in the pH of the leaching fluid to pH-neutral is further explained.

5 Sample material/test piece

For the single execution of the test according to this standard a sample of 0,85 l ± 0,1 l of granular material is necessary with a maximum grain size (95 %) of 4 mm at the most. Coarse-grained materials and moulded and monolithic waste materials shall first be crushed.

Of the available sample 0,8 l ± 0,1 l shall be processed into a test piece P by compacting the material in a cylindrical vessel (7.1), saturating it with water (6.1) and covering it with a layer of glass beads (7.2) with a thickness of 1 cm ± 0,2 cm.

NOTE 1 If the diffusion test is carried out (partly) to determine the effective diffusion coefficient and/or the leaching per mass unit, an extra test piece is necessary to carry out the availability test according to NEN 7371. The mass (m) in kg and the density (ρ) in kg/m^3 of test piece P then also has to be known.

NOTE 2 Standards are available for the sampling of solid earthy and stony building and waste materials for leaching tests. It is recommended that the procedures described in NEN 7300, [2] and NEN-EN 14899 be used.

NOTE 3 If the sample from which the test piece P is obtained has to undergo pretreatment, it is recommended that the procedures described in NEN 7310 be used.

6 Reagents

6.1 Demineralized pH-neutral water

Demineralized pH-neutral water with a conductivity of max. 1 µS/cm.

6.2 Nitric acid of analytically pure quality, 1 mol/l

7 Equipment and requisites

The equipment and requisites listed below shall be checked before use for proper operation and absence of disrupting elements that may affect the result of the test. They may not give off or absorb any of the components to be determined in the eluates.

The equipment listed under 7.6 and 7.7 shall also be calibrated.

7.1 Sealable outer vessel with cylindrical inner vessel

Sealable outer vessel with a cylindrical inner vessel, both of plastic without softeners, in which the material to be tested can be compacted and exposed on one side to a leaching fluid. The inner vessel shall have an internal diameter of 10 cm ± 0,5 cm and an internal height of 10 cm ± 0,5 cm. The outer vessel shall be of such dimensions that during the test for each refreshment cycle no more than 0,75 l leaching fluid is necessary to allow the fluid level to end up at least 2 cm above the inner vessel.

The outer vessel shall be fitted at the bottom with a tap in such a way that during the different refreshment cycles eluates can be drawn off without disrupting the diffusion profile in the test piece P.

NOTE 1 For an explanatory note on the construction of the test set-up see A.1 and figure A.1.

NOTE 2 The dimensions of the inner and outer vessel are selected such that for the materials tested measurable concentrations are usually obtained in the eluates (see A.4).

NOTE 3 The height of the inner vessel is selected such that in the material to be tested no exhaustion of any component can occur during the course of the test.

7.2 Glass beads

Glass beads with a diameter between 2 mm and 3 mm.

7.3 Filtration equipment

Filtration equipment, suitable for filtration at an increased or reduced pressure.

7.4 Membrane filters

Not previously used membrane filters for the filtration equipment (7.3) with a pore size of 0,45 µm.

7.5 Collection bottles

Sealable plastic collection bottles.

7.6 pH meter

pH meter calibrated according to NEN 6411 with a measurement accuracy better than ± 0,05 pH units.

7.7 Conductivity meter

Conductivity meter calibrated according to NEN-ISO 7888 with a measurement accuracy better than ± 1 %.

7.8 Graduated beaker or balance

A graduated beaker with a measurement range up to at least six times the volume V_p of the test piece P and a measurement accuracy better than ± 1 %. Or a balance with a measurement range up to at least three times the mass of the test piece P and a measurement accuracy better than ± 0,1 %.

8 Procedure

8.1 General

The leached quantities and the diffusion coefficient that determines the degree of leaching are determined by, in succession:

— determining the requirements for the eluate samples to be analysed according to 8.2;

— carrying out the granular diffusion test according to 8.4;

— analysing the eluates according to 8.5.

8.2 Eluate samples

Determine the quantity of eluates that is necessary for the analysis of the leached components and determine the way in which the eluate samples shall be stored according to the following procedure:

a) check how many analyses shall be carried out, for which components and according to which methods;

b) check for which components the eluates shall be preserved and in what way;

c) determine in the light of the above for each component to be analysed the minimum necessary quantity of eluates and the way in which the eluate samples shall be preserved and stored. For this, use the guidelines for preservation of surface and wastewater from NEN-EN-ISO 5667-3.

NOTE When carrying out the single granular diffusion test, approx. 750 ml of eluate sample is available for chemical analysis.

8.3 Preparation of the test piece P

Determine the area A of the internal cylinder diameter of the inner vessel (7.1).

Rinse the outer and inner vessel (7.1) before carrying out the test with nitric acid (6.2) and rinse afterwards with demineralized water (6.1).

The inner vessel shall be filled as follows.

Weigh the vessel empty (M_{ves1}) with an accuracy of ± 10 mg (7.8).

Weigh with the same accuracy a quantity of material that is necessary as a minimum to completely fill the inner vessel (M_{m1}). Weigh with an accuracy of ± 10 mg an excess of demineralized water (6.1) that is necessary as a minimum to fill the outer vessel to 2 cm above the rim of the inner vessel (M_{w1}). Place a 1 cm to 2 cm thick layer of the weighed quantity of material on the bottom of the inner vessel and saturate this layer with part of the quantity of water weighed. Compact the layer by dropping a drop weight of approx. 200 g four times from a height of approx. 20 cm onto the material. Add another layer of 1 cm to 2 cm, saturate this layer too with part of the quantity of water weighed and compact the layer with the drop weight. Repeat this layer by layer build-up until the inner vessel is filled to 1 cm ± 0,2 cm below the rim.

Weigh the vessel thus filled (M_{ves2}), the remainder of the excess material (M_{m2}) and the remainder of the quantity of water weighed (M_{w2}).

Calculate from the dimensions of the vessel the volume of the test piece (V_P). Calculate the dry density of the material in the inner vessel (ρ) according to:

$$\rho = (M_{m1} - M_{m2})/V_P \qquad (1)$$

where:

ρ is the dry density of the material in the test piece P;

M_{m1} is the weighed quantity of material with which the filling procedure of the inner vessel was started;

M_{m2} is the remainder of the weighed quantity of material M_{m1} after the end of the filling procedure;

V_P is the volume of the test piece P.

The difference between ($M_{m1} - M_{m2} + M_{w1} - M_{w2}$) and $M_{ves2} - M_{ves1}$ shall not be more than 5 %, where:

M_{w1} is the weighed quantity of demineralized pH-neutral water with which the filling procedure of the inner vessel was started;

M_{w2} is the remainder of the weighed quantity of water M_{w1} after the filling procedure;

M_{ves1} is the mass of the empty inner vessel;

M_{ves2} is the mass of the inner vessel after the filling procedure.

Place a layer of glass beads (7.2) with a height of 1 cm ± 0,2 cm on the water-saturated layer of material and place the inner vessel in the outer vessel.

8.4 Execution of the granular diffusion test

The diffusion test is carried out in eight steps at a temperature that may vary between 18 °C and 22 °C.

8.4.1 Step 1

Fill the outer vessel with demineralized pH-neutral water (6.1).

The water level shall be at least 2 cm above the rim of the inner vessel.

Close the outer vessel.

Draw off all the eluates after 6 h ± 0,5 h. This is the eluate fraction from period 1. Determine the volume *V* of this fraction to an accuracy of 2 %.

Filter a quantity of eluates intended for analysis according to the instructions of 8.2 over a membrane filter (7.3 and 7.4).

Measure the pH (± 0,05) and the conductivity (± 1 mS/cm) of the eluates thus obtained.

NOTE The value of the pH says something about the alkalinity of the test piece. The course of the pH during the granular diffusion test gives an indication of the stability of the material tested. Great variations in the pH of the eluates indicate that the material tested is not yet in equilibrium, or is not yet stable. The conductivity gives information on the salt load in the eluates, which is important for the analysis.

Transfer the quantities of eluates intended for the analysis into suitable bottles (7.5), where each bottle is filled with at least 10 ml.

Preserve the eluate samples according to the procedure required in 8.2.b. If more than 1 ml of preservative is necessary per 250 ml eluates, the concentrations determined according to 8.5 shall be corrected for this.

8.4.2 Step 2 to 8

Immediately after drawing off according to step 1 refill the vessel with demineralized pH-neutral water (6.1). Use to an accuracy of 2 % the same quantity *V* as drawn off in step 1.

Repeat the procedure described in step 1 a further seven times according to the schedule in Table 1 (the times apply from submersion).

Note to an accuracy of 15 min the refreshment times (time when the tray is just emptied) of each period *n*.

8.5 Analysis

Analyse the eluate fractions obtained according to 8.4 from periods 1 to 8.

Analyse the eluate samples within the periods indicated in the guideline according to NEN-EN-ISO 5667-3.

NOTE Standards are available for the chemical analysis of eluates for a number of components. It is recommended that the procedures described in NEN 6952, [3] and [12] be used.

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