Leaching characteristics of solid earthy and stony building and waste materials.

Leaching tests. Determination of the leaching of inorganic components from granular materials with the column test

Uitloogkarakteristieken van vaste grond- en steenachtige bouwmaterialen en afvalstoffen. Uitloogproeven. Bepaling van de uitloging van anorganische componenten uit poeder- en korrelvormige materialen met de kolomproef

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Introduction

In order to determine the various aspects of the leaching behaviour (leaching characteristics) of solid earthy and stony building and waste materials, a number of stages must be carried out namely sampling, sample pretreatment, leaching tests, digestion and chemical analysis of the solid substance and eluate respectively. Each stage has its own coordinating standard which gives general instructions. This document specifies the correlation between all standards relating to the stage with a specific area of application for each. For the determination of the leaching characteristics, the general instructions and the specific standards cited shall be followed in close mutual correlation.

The general instructions for the choice and applicability of the leaching tests are described in NEN 7340; this general standard also specifies to which type of materials and which components the present standard can be applied. NEN 7340 also gives definitions of the specific terms used in NEN 7343.

NEN 7343 describes the column test, with which the fraction available for leaching is determined, as a function of the ratio between liquid and solid (the value of L/S), over a range varying from 0.1 l to 10 l per kg dry matter. The value of L/S can be related to a timescale, so that based on the results of the column test an opinion can be formed on the time-dependence of the leaching of a material under practical conditions.

The column test was previously described as part of NVN 2508. By the publication of NEN 7343 (and NEN 7349) NVN 2508 is withdrawn.

The standards which characterize the various aspects of the leaching behaviour are produced and published in phases. This means that on publication of NEN 7343, some of the relevant standards will not yet have appeared as drafts or standards. For the missing stages, users of NEN 7343 must select the method to be applied. The standards listed in annex C and other publications available in this context may also be used.

The numbered chapters are normative with the exception of the passages marked "NOTE"; the introduction and appendices are given for information only.
1 Scope

This standard describes the column test to determine the leaching of inorganic components from solid earthy and stony materials and wastes as a function of the value of L/S, over a range varying from 0.1 l to 10 l per kg of dry matter.

For an itemized list of the materials with which experience has been acquired, with the execution of the column test and/or for which the applicability of the column test has been tested, in a round-robin test according to NEN-ISO 5725 and for which the precision in terms of repeatability and reproducibility is described, reference is made to NEN 7340 and annex A.

NOTE

To determine the leaching of inorganic components from solid (stony and earthy) materials as a function of the value of L/S, NEN 7340 refers to two methods each with its own range of values of L/S. The column test according to NEN 7343 covers the values of L/S from 0.1 l to 10 l per kg dry matter, the cascade test according to NEN 7338 is intended for values of L/S of 20 l to 100 l per kg dry matter.

2 Normative references


NEN 7312:1995 Leaching characteristics of solid earthy and stony building materials and waste products. Sample pretreatment. Sample pretreatment for determination of the leaching behaviour and the concentration of inorganic components.


NPR 6598:1992 Water. Rounding of analysis results

NEN-ISO 5725:1987 Precision of testing methods. Determination of the repeatability and reproducibility of standardized test methods by inter-laboratory testing.

3 Terms and definitions

For the definitions of the terms used in this standard reference is made to clause 3 of NEN 7340:1995 and to clause 3 of NEN 7310:1995.

4 Principle

The aim of the column test is to simulate the leaching of inorganic components from powdered and granular materials in an aerobic environment as a function of the value of L/S over a range varying from 0.1 l to 10 l per kg of dry matter.

In the column test the leaching liquid (acidified water) is passed through the material to be tested in a vertical column from the bottom, whereby after set quantities of leaching liquid have been passed through it, the concentrations of the leached components in the eluate are measured. The pH value of the eluate is enforced by the material itself. Based on the column test results the leached quantity can be calculated for each component analyzed both per fraction collected and cumulatively.

5 Samples for analysis

To carry out a single column test, between 0.5 l and 0.7 l of sample for analysis is needed, for which the moisture content g is known and for which at least 95 % (m/m) (dry matter) of the particles are smaller than 4 mm.

NOTE

1. Standards are being prepared for leaching tests for sampling solid earthy and stony building and waste materials. It is recommended that the methods described in NEN 7300 and [1] be used.

2. Where the sample from which the sample for analysis is obtained shall undergo pretreatment, it is recommended that the methods described in NEN 7310 should be used.

The moisture content g of the sample for analysis shall be determined on a separate sub-sample that is dried at (105 ± 5) °C in accordance with NVN 7312.

NOTE

Where the sample intended for the column test has to be pre-dried to prepare the sample for analysis, this may not be carried out at a temperature higher than 40 °C to prevent components evaporating or chemical conversions taking place during the drying process which affect the leaching behaviour. If the material already has the right grain size distribution, no pre-drying should be carried out.

6 Reagents

6.1 Demineralized water with a conductivity of a maximum of 1 μS/cm, acidified with nitric acid of analytically pure quality to pH = 4 ± 0.1.

6.2 Nitric acid of analytically pure quality (HNO₃) = 1 ± 0.1 mol/l.

7 Apparatus

The materials and equipment mentioned below shall be checked before use for proper operation and absence of interfering elements which may affect the result of the test.

7.1 Column with an internal diameter of (5 ± 0.5) cm and a fillable height of at least 4 times the internal diameter, fitted with shut-off valves in which filters (7.2 and 7.3) can be fitted.

7.2 Membrane filters for the column with a pore size of 0.45 μm.

7.3 Prefilters for the column with a pore size of a maximum of 1.5 μm.

7.4 Peristaltic pump with an adjustable and readable capacity of between 0 ml/h and 50 ml/h.

7.5 Polyethylene collection flasks with screwcap.

7.6 Analytical balance with a measurement accuracy better than ± 10 mg.

7.7 pH meter with a measurement accuracy better than ± 0.05 pH units.
8 Procedure

Leaching as a function of the value of \( L/S \) is determined by successively:

- determining the requirements of the eluate samples to be analyzed in accordance with 8.1;
- carrying out the column test in accordance with 8.2;
- analyzing the eluate in accordance with 8.3;
- carrying out the calculations in accordance with clause 9.

8.1 Eluate samples

Determine the quantity of eluate needed to analyse the leached components and the way in which the eluate samples shall be stored in accordance with the following procedure:

a) first check how much, for what components and by what methods analyses shall be carried out;

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

c) determine in the light of the above the minimum quantity of eluate necessary for each component to be analyzed and the way in which the eluate samples shall be preserved.

NOTE
To prevent precipitation or evaporation of certain components, the eluate should be preserved. Preservation of metals in the eluate is usually carried out by acidification with nitric acid to \( \text{pH} = 2 \) (for an hydrochloric acid is used; mercury is preserved by adding nitric acid and potassium dichromate) to preserve anions (for example chloride, sulphate, fluoride), no acidification should be carried out.

8.2 The column test

The column test is carried out in seven stages at a temperature which may vary between 18 °C and 22 °C.

8.2.1 Step 1

Rinse column (7.1), supply and outlet hoses (7.9), filters (7.2 and 7.3) and collection flasks (7.5) with nitric acid (6.2) before the start of the test and afterwards rinse with acidified water (6.1). Weigh the column, including valves and filters to an accuracy of 10 mg.

Fill the column with the sample to be analyzed (5) up to a bed height of at least four times the internal diameter of the column. During filling care shall be taken to ensure a good packing in the column. If necessary use a vibration plate. Fit valves, equipped with membrane filters (7.2), to the bottom and top of the column to prevent entrainment of fine particles with the eluate.

Where the grain size distribution of the sample for analysis is such that the membrane filter (7.2) may clog, a pre-filter (7.3) shall also be used. The valves must be fitted such that the liquid flow cannot bypass the filters and such that no open space is left above the material.

Weigh the column thus filled to an accuracy of 10 mg.

Determine the dry mass in the column of the sample for analysis in accordance with:

\[
m_0 = m(1 - g)
\]  

where:
- \( m_0 \) is the mass of the sample for analysis in the column, in kg dry matter;
- \( m \) is the quantity of sample for analysis determined by weighing, in kg;
- \( g \) is the moisture content according to NVN 7312, in g/kg.

Connect pump (7.4) to the bottom of the column and pass acidified water through the column during the test from bottom to top until it is saturated, followed by a total quantity of \((10 \pm 0,02) m_0 \) (6.1). Connect the top of the column to a collection flask (7.5).

Set the flow rate \( q \) of the pump to a value calculated in accordance with:

\[
q = a \times m_0
\]

where:
- \( q \) is the flow rate of the pump in l/h;
- \( m_0 \) is the mass of the sample for analysis in the column, in kg dry matter;
- \( a \) is a coefficient equal to 0,025 l/kg · h.

NOTE
At the maximum permitted pump flow rate the column test takes approx. three weeks. When using sludges and clayey materials with a low permeability the test may be carried out at an increased differential pressure. Where the maximum permitted flow rate still cannot be reached, the test will last longer.

Replace the collection flask (7.5) connected with a new one as soon as a quantity of \((0,1 \pm 0,01) m_0 \) l of acidified water (6.1) has passed through after the column is completely saturated. This is the first fraction \((k_1)\).

Measure the pH \((\pm 0,1)\) and conductivity \(K_{20} \pm (1 \mu S/cm)\) of the eluate collected. Transfer the quantities of eluate intended for analysis into suitable flasks (7.5), but fill each flask with at least 10 ml. Preserve the eluate samples according to the procedure described in 8.1. Where more than 1 ml of preservation fluid is needed per 250 ml of eluate, the concentrations determined according to 8.3 shall be corrected for this.

8.2.2 Steps 2 to 7

Change the collection flask with a new one as soon as a quantity of acidified water (6.1) according to table 1 has been passed through. These are fractions \(k_2\) to \(k_7\).

NOTE
The symbol \( k \) in the fractions stands for “column test” and the index 1 to 7 indicates the serial number.

Measure the pH \((\pm 0,1)\) and conductivity \((\pm 1 \mu S/cm)\) of the eluate fractions collected and keep the preserved eluate fractions in separate sealed flasks.

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Fraction Volume</th>
<th>( L/S )</th>
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</thead>
<tbody>
<tr>
<td>( k_1 )</td>
<td>((0,1 \pm 0,01) m_0 )</td>
<td>((0,1 \pm 0,01) )</td>
</tr>
<tr>
<td>( k_2 )</td>
<td>((0,1 \pm 0,01) m_0 )</td>
<td>((0,2 \pm 0,02) )</td>
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<tr>
<td>( k_3 )</td>
<td>((0,3 \pm 0,03) m_0 )</td>
<td>((0,5 \pm 0,05) )</td>
</tr>
<tr>
<td>( k_4 )</td>
<td>((0,5 \pm 0,05) m_0 )</td>
<td>(1,0 \pm 0,1 )</td>
</tr>
<tr>
<td>( k_5 )</td>
<td>((1,0 \pm 0,1) m_0 )</td>
<td>((2,0 \pm 0,2) )</td>
</tr>
<tr>
<td>( k_6 )</td>
<td>((3,0 \pm 0,3) m_0 )</td>
<td>((5,0 \pm 0,5) )</td>
</tr>
<tr>
<td>( k_7 )</td>
<td>((5,0 \pm 0,5) m_0 )</td>
<td>((10,0 \pm 0,02) )</td>
</tr>
</tbody>
</table>
8.3 Analysis

Analyse the eluate samples obtained in accordance with 8.2 as quickly as possible after each stage and as far as possible in one series.

NOTE
Standards are being prepared for the chemical analysis of eluates for a number of components. It is advised that provisional use be made of the methods described in NEN 7320 and [2].

9 Calculation

Calculate for each component the leached quantities in all eluate fractions with the formula:

\[ \frac{U_{col,i}}{m_0 \times c_i \times f} \]

where:
- \( i \) is the index of the eluate fraction (1, 2, ..., 7);
- \( U_{col,i} \) is the leached quantity of a component per quantity of sample for analysis in mg per kg dry matter, in the eluate fraction \( i \) of the column test;
- \( c_i \) is the concentration of that component in the eluate fraction \( i \), in g/l;
- \( m_0 \) is the mass of the sample for analysis in the column in kg dry matter;
- \( V_i \) is the volume of the eluate fraction \( i \) in l;
- \( f \) is a dimensionless factor: 1000 mg/g.

The concentration \( c_i \) referred to in formula (3) is the concentration originally present in the eluate, the measured value determined in accordance with 8.3 shall be corrected for the quantity of preservation fluid added in 8.2 where this is more than 1 ml per 250 ml eluate.

Where the concentration of a component in one or more eluate fractions is below the lower detection limit, two calculations shall be carried out for this component in these fractions. The upper limit of \( U_{col,i} \) is calculated by making \( c_i \) in formula (3) equal to the lower detection limit; the lower limit of \( U_{col,i} \) is calculated by making \( c_i \) in formula (3) equal to 0.

Also calculate for each component the cumulative leaching (\( \Sigma U_{col,i} \)) by adding up the leached quantities of the relevant component in the following eluate fractions collected. Where the concentration of a component in a certain eluate fraction is below the lower detection limit, for this component two calculations shall be carried out, to indicate both the upper limit and the lower limit of \( \Sigma U_{col,i} \).

NOTE
The leaching per component may also be expressed as a percentage of the original content of the relevant component in the sample for analysis. For this reference is made to annex B.

10 Report

The report shall contain at least the following data:
- a reference to this standard indicating: " in accordance with NEN 7343:1995";
- the data needed to identify the sample for analysis;
- the origin and specifications of the sample for analysis;
- the nature of the material examined;
- the temperature range within which the leaching test has been carried out;
- the duration of the test where this is more than three weeks;
- the values of L/S obtained in the column test, rounded to a maximum of two significant figures according to NPR 6598;
- the pH of the eluates collected, rounded to 0.1 pH unit;
- the conductivity of the eluates collected, rounded to a maximum of one significant figure according to NPR 6598;
- the components which are analyzed and the lower detection limits of these components in the eluate;
- all measured concentrations, rounded to a maximum of two significant figures according to NPR 6598;
- the quantity of preservation fluid added in accordance with 8.2 where this is more than 1 ml per 250 ml eluate;
- the leached quantity \( U_{col,i} \) calculated per fraction for each component, in mg per kg dry matter, rounded to a maximum of two significant figures according to NPR 6598; for analysis results below the lower detection limit for the relevant component both the lower limit and the upper limit of the leached quantity shall be indicated;
- the cumulative leached quantity of \( \Sigma U_{col,i} \) calculated for each component, in mg per kg dry matter, rounded to a maximum of two significant figures according to NPR 6598; in the case of analysis results below the lower detection limit for the relevant component both the upper limit and the lower limit of \( \Sigma U_{col,i} \) shall be indicated;
- the date of the test.

Where the column test is not carried out fully in accordance with this standard, reference may only be made to NEN 7343:1995 in the report in case all deviations from the procedures prescribed in this standard are indicated in the report stating the reasons for deviation.
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