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Characterization of waste — Leaching behaviour test — Influence of pH on leaching with initial acid/base addition

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National foreword

This British Standard is the UK implementation of EN 14429:2015. It supersedes DD CEN/TS 14429:2005 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/508/3, Characterization of waste.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Characterization of waste - Leaching behaviour test - Influence of pH on leaching with initial acid/base addition

Caractérisation des déchets - Essais de comportement à la lixiviation - Influence du pH sur la lixiviation avec ajout initial d'acide/base

Charakterisierung von Abfällen - Untersuchung des Elutionsverhaltens - Einfluss des pH-Wertes auf die Elution unter vorheriger Säure/Base-Zugabe

This European Standard was approved by CEN on 26 December 2014.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Contents

Page

Foreword

This document (EN 14429:2015) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2015, and conflicting national standards shall be withdrawn at the latest by September 2015.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 14429:2005.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

The following significant technical changes have been implemented in this new edition of the text:

— the status of the document has been changed from a CEN/TS into a European Standard;

— performance data has been added (see Annex E).

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This document has been developed primarily to support the requirements for leaching behaviour testing within EU and EFTA countries.

This document specifies a test method to determine the influence of pH on the leachability of inorganic constituents from waste materials.

For the complete characterization of the leaching behaviour of waste under specified conditions the application of other test methods is required (see EN 12920).

Anyone dealing with waste and sludge analysis should be aware of the typical risks of that kind of material irrespective of the parameter to be determined. Waste and sludge samples can contain hazardous (e.g. toxic, reactive, flammable, infectious) substances, which can be liable to biological and/or chemical reaction.

Consequently these samples should be handled with special care. Gases which can be produced by microbiological or chemical activity are potentially flammable and will pressurize sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. National regulations will be followed with respect to all hazards associated with this method.

In the different European countries, tests have been developed to characterize and assess the constituents which can be leached from waste materials. The release of soluble constituents upon contact with water is regarded as one of the main mechanism of release which results in a potential risk to the environment during life-cycle of waste materials (disposal or re-use scenario). The intent of these tests is to identify the leaching properties of waste materials. The complexity of the leaching process makes simplifications necessary. Not all of the relevant aspects of leaching behaviour can be addressed in one single standard.

Procedures to characterize the behaviour of waste materials can generally be divided into three steps, using different tests in relation to the objective. The following test hierarchy is taken from the Landfill Directive [1)] and the Decision on Annex II of this Directive [2)] for disposal of waste.

a) Basic characterization constitutes a full characterization of the waste by gathering all the necessary information for a safe management of the waste in the short and long term. Basic characterization may provide information on the waste (type and origin, composition, consistency, leachability, etc.), information for understanding the behaviour of waste in the considered management scenario, comparison of waste properties against limit values, and detection of key variables (critical parameters as liquid/solid (*L*/*S*) ratios, leachant composition, factors controlling leachability such as pH, redox potential, complexing capacity and physical parameters) for compliance testing and options for simplification of compliance testing. Characterization may deliver ratios between test results from basic characterization and results from simplified test procedures as well as information on a suitable frequency for compliance testing. In addition to the leaching behaviour, the composition of the waste should be known or determined by testing. The tests used for basic characterization should always include those to be used for compliance testing.

b) Compliance testing is used to demonstrate that the sample of today fits the population of samples tested before by basic characterization and through that, is used to carry out compliance with regulatory limit values. The compliance test should therefore always be part of the basic characterization program. The compliance test focuses on key variables and leaching behaviour identified by basic characterization tests. Parts of basic characterization tests can also be used for compliance purposes.

1) Council Directive 1999/31/EC of 26 April 1999 on the landfill of waste.

2) Council Decision 2003/33/EC of 19 December 2002.

c) On-site verification tests are used as a rapid check to confirm that the waste is the same as that which has been subjected to characterization or compliance tests. On-site verification tests are not necessarily leaching tests.

The test procedure described in this document is a basic characterization test and falls in category a).

In this European Standard leaching is carried out at different pH values as a result of the reaction between pre-selected amounts of acid or base and test portions of the waste material. Size reduction is performed to facilitate approaching equilibrium.

This test is different from the "pH dependence test with continuous pH control" (pH static test, see EN 14997) in which the pH is controlled at pre-selected values over the entire testing period by continuous measurement and automatic addition of acid or base. The test is aiming at approaching equilibrium at the end of the procedure.

NOTE In Annex B specific uses of both the pH dependence test with initial acid/ base addition and the pH dependence test with continuous pH control are indicated.

1 Scope

This European Standard specifies a method for the determination of the influence of pH on the leachability of inorganic constituents from a waste material. The equilibrium condition as defined in the standard is established by addition of pre-determined amounts of acid or base to reach desired end pH values. This test method produces eluates, which are subsequently characterized physically and chemically.

This European Standard is a parameter specific test as specified in EN 12920. The application of this test method alone is not sufficient for the determination of the detailed leaching behaviour of a waste under specified conditions.

2 Normative references

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

EN 14346:2006, *Characterization of waste — Calculation of dry matter by determination of dry residue or water content*

EN 14899, *Characterization of waste — Sampling of waste materials — Framework for the preparation and application of a Sampling Plan*

EN 15002, *Characterization of waste — Preparation of test portions from the laboratory sample*

EN 16192, *Characterization of waste — Analysis of eluates*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

EN ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples (ISO 5667-3)*

3 Terms and definitions

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

3.1
dry residue
w_{dr}
remaining mass fraction of a sample after a drying process at 105 °C

[SOURCE: EN 14346:2006]

3.2
eluate
solution obtained by a leaching test

3.3
equilibrium
condition achieved when the pH deviation during a checking period at the last 4 h of the test is below 0,3 pH unit

3.4
laboratory sample
sample or subsample(s) sent to or received by the laboratory

[SOURCE: IUPAC, 1990]

Note 1 to entry: When the laboratory sample is further prepared (reduced) by subdividing, cutting, crushing, sawing, coring, or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test or for analysis. The laboratory sample is the final sample from the point of view of sampling but it is the initial sample from the point of view of the laboratory.

Note 2 to entry: Several laboratory samples may be prepared and sent to different laboratories or to the same laboratory for different purposes. When sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

3.5
leachant
liquid that is brought into contact with the test portion in the leaching procedure

3.6
liquid to solid-ratio
L*/*S
ratio between the amount of liquid (*L*) and of solid (*S*) in the test

Note 1 to entry: *L*/*S* is expressed in l/kg dry matter.

3.7
suspension
mixture of leachant and test portion

3.8
test portion
amount or volume of the test sample taken for analysis, usually of known weight or volume

[SOURCE: IUPAC, 1990]

3.9
test sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or analysis

[SOURCE: IUPAC, 1990]

4 Symbols and abbreviations

For the purposes of this document, the following symbols and abbreviations apply.

ANC acid neutralization capacity

BNC base neutralization capacity

DM dry matter

DOC dissolved organic carbon

L/*S* liquid to solid-ratio

M_d dried mass of the test portion

m_d mass after drying

M_w un-dried mass of the test portion

m_r mass before drying

t_0 time at the start of the leaching test

$V_{A/B}$ volume of acid or base used in leachant

V_{demin} volume of demineralized water used in leachant

V_L volume of added leachant

w_{dr} dry residue of the sample

5 Principle

This European Standard describes a method to determine the influence of pH on the leachability of inorganic constituents from a waste material.

Separate test portions are leached at a fixed *L*/*S* ratio with leachants containing pre-selected amounts of acid or base in order to reach stationary pH values at the end of the extraction period. Each leachant is added in three steps in the beginning of the test. At least 8 final pH values are required, covering at the minimum the range pH 2 to pH 12 (both included i.e. the lowest value ≤ 2 and the highest value ≥ 12). The amounts of acid or base needed to cover the pH range can be derived from the results of a preliminary titration, from available experimental data on the material to be tested or from an arbitrary division of the predetermined maximum consumption of acid and base. The tests are carried out at a fixed contact time at the end of which equilibrium can be assumed to be approached for most constituents in most waste materials to be characterized. The approaching of equilibrium as defined in the standard is verified at the end of the extraction period.

The results are expressed in mg/l of constituents for each final pH value. For each final pH value also the quantity of acid that is added is expressed in mol H^+/kg DM and the quantity of base that is added is expressed as mol OH^-/kg DM (for graphical presentation mol OH^-/kg DM is expressed as – mol H^+/kg DM).

NOTE Other expression of results is possible (including mg leached /kg dry matter). Since this test is aiming at approaching equilibrium i.e. solubility controlled, the results alone cannot be used to quantify the soluble mass fraction.

The acid or base neutralization capacity (ANC, BNC) of the waste is also determined.

The pH range covered by the test may be restricted to a pH range relevant for the specific material and the considered problem (see 9.2).

6 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

6.1 Distilled water, demineralized water, de-ionized water or water of equivalent purity (5 < pH < 7,5) with a conductivity < 0,1 mS/m according to grade 2 specified in EN ISO 3696.

6.2 Nitric acid, $c(HNO_3)$ = 0,1 mol/l to 5 mol/l.

6.3 Sodium hydroxide, $c(NaOH)$ = 0,1 mol/l to 5 mol/l.

Sodium hydroxide is unstable due to possible uptake of CO_2. Therefore it is recommended to prepare a fresh solution.

7 Equipment

7.1 General

Check the materials and equipment specified in 7.2.1, 7.2.4 and 7.2.9 before use for proper operation and absence of interfering elements that may affect the result of the test.

Calibrate the equipment specified in 7.2.2, 7.2.6, 7.2.7 and 7.2.8.

7.2 Laboratory equipment

Usual laboratory apparatus, and in particular the following:

7.2.1 Agitation device, end-over-end tumbler (5 r/min to 10 r/min) or roller table rotating at about 10 r/min.

7.2.2 Analytical balance, with an accuracy of at least 0,1 g.

7.2.3 Glass or plastic bottles, e.g. high density polyethylene (HDPE)/polypropylene (PP)/polytetrafluoroethylene (PTFE)/polyethyleneterephtalate (PET).

Use bottles with an appropriate volume (250 ml for the test portions of 15 g of dry mass, 500 ml for the test portions of 30 g of dry mass and 1 l for the test portions of 60 g of dry mass), and with screw caps.

For eluate collection and preservation of eluate samples use bottles with an appropriate volume and with screw caps (rinsed in accordance with EN ISO 5667-3).

7.2.4 Crushing equipment, jaw crusher or a cutting device.

NOTE Crushing is prescribed to avoid unnecessary grinding to very fine particle sizes, such as takes place in a rotary swing mill, ball mill or similar device.

7.2.5 Membrane filters, with a pore size of 0,45 µm.

Membrane filters for the filtration device, fabricated from inert material, which is compatible with the waste. Filter shall be pre-rinsed with demineralized water or similarly clean in order to remove DOC.

7.2.6 pH meter, with an accuracy of at least 0,05 pH units.

7.2.7 Conductivity meter, with an accuracy of at least 0,1 mS/m.

7.2.8 Redox potential meter (optional).

7.2.9 Vacuum filtration device or pressure filtration device.

7.2.10 Sieving equipment, with sieve of 1 mm nominal screen sizes.

Due to crushing and sieving, contamination of the sample may occur to an extent, which may affect the leached amounts of some constituents of concern, e.g. Co and W from tungsten carbide crushing equipment or Cr, Ni, Mo and V from stainless steel equipment.

8 Sample preparation

8.1 Laboratory sample

The laboratory sample shall consist of a mass equivalent of at least 1 kg of dry mass. In case less material is available, a justification shall be provided in the test report.

Perform sampling in accordance with EN 14899 or a standard derived from EN 14899 in order to obtain a representative laboratory sample.

8.2 Preparation of the test sample

The tests shall be made on material with a grain size of 95 % less than 1 mm. In order to ensure that the test sample consists in 95 % mass of particles less than 1 mm in diameter, it shall be sieved, using the sieving equipment (see 7.2.4), to separate the oversized particles. If oversized material exceeds 5 % (mass), the entire oversized fraction shall be crushed. Any non-crushable material (e.g. metallic parts such as nuts, bolts, scrap) shall be separated from the oversized fraction and the weight and nature of the non-crushable material shall be recorded. Crushed and uncrushed material shall be mixed to constitute the test sample.

Moist material that is not possible to sieve needs to be dried prior to sieving and/or crushing. The drying temperature shall not exceed 40 °C.

Perform size reduction, drying, if needed, and sub-sampling according to specifications provided in EN 15002.

NOTE 1 There is no obligation to sieve if it is obvious that the material will fully pass the sieve on 1 mm.

NOTE 2 The crushed material can change upon storage due to ageing of fresh surfaces. It is therefore preferable to test the material as soon as possible after crushing.

It is recommended that materials with a high natural pH be crushed under nitrogen in order to avoid contact with air leading to carbonation.

8.3 Determination of dry residue

The whole test sample, complying with the size criteria in 8.2 shall not be further dried. The dry residue (w_{dr}) of the test sample shall be determined on a separate test portion.

The dry residue shall be determined at 105 °C ± 3 °C according to EN 14346. The dry residue is calculated as follows:

$$w_{dr} = 100 \times \frac{m_d}{m_r} \tag{1}$$

where

w_{dr} is the dry residue of the waste, expressed as a percentage (%);

m_d is the mass after drying, in grams (g);

m_r is the mass before drying, in grams (g).

8.4 Preparation of the test portion

Prepare at least 8 test portions by the use of a sample splitter or by coning and quartering in accordance with EN 15002. Based on sample heterogeneity and eluate volume requirement for analysis, test portion size shall be either M_d = 15 g, 30 g or 60 g (with a tolerance of ± 10 %).

Calculate the undried mass of the test portion M_w to be used for the test as follows:

$$M_{w} = \frac{M_{d}}{w_{dr}} \times 100 \tag{2}$$

where

M_w is the undried mass of the test portion, in grams (g);

M_d is the dried mass of the test portion, in grams (g);

w_{dr} is the dry residue of the waste, expressed as a percentage (%).

9 Procedure

9.1 Contact time

The leaching procedure consists of three defined stages:

— Period A (acid/base addition) from t_0 up t_0 + 4 h for acid/base addition in three steps;

— Period B (equilibration period) from t_0 + 4 h up to t_0 + 44 h equilibration period;

— Period C (verification period) from t_0 + 44 h up to t_0 + 48 h for verification of equilibrium condition.

Measure the pH in the liquid after each of these periods.

The total contact period (A+B+C) is 48 h.

9.2 pH range

The test shall cover the range pH 2 to pH 12 (both included i.e. the lowest value ≤ 2 and the highest value ≥ 12) with at least 8 pH values tested including the natural pH (without acid or base addition). The maximum difference between two consecutive pH values shall not exceed 1,5 pH units.

NOTE To ensure that the appropriate pH values can be obtained in one run additional bottles can be prepared of which only the ones with the desired final pH values are retained for analysis.

The pH range covered by the test may be restricted to a pH range relevant for the specific material and the considered problem. The pH range to be covered can also depend on the specific properties of the waste material, the available information on this material and the questions to be answered by performing the test. The number of pH levels considered can be reduced or increased in a specific pH domain as needed. Release measured at low pH (pH = 2) can be used to estimate the potential availability for leaching, which is a relevant property for geochemical modelling.

9.3 Leaching test

9.3.1 General

The following procedure applies for each of the chosen pH values to be tested.

9.3.2 Preparation of leachant

Estimate the acid or base consumption for reaching the relevant pH values. Symbol A (mol H^+/kg dry matter) is used for the pre-estimated acid consumption and symbol B (mol OH^-/kg dry matter) is used for the pre-estimated base consumption. Use this to determine the required acid and base strength.

The acid or base consumption for the considered pH values may be derived from available information, from the preliminary procedures in Annex C or from information in Annex D.

Calculate the volume V of liquid to establish L/S = 10 l/kg ± 0,2 l/kg for the actual size of test portion M_w (see 8.4) including the volume of acid or base.

$$V = 10 \times M_d \tag{3}$$

where

V is the total volume of liquid in the test, in ml;

M_d is the dry mass of the test portion, in grams (g) (see 8.4).

NOTE In relation to L/S-ratio, V in this equation is equivalent with the "L" and M_d is equivalent to the "S".

Calculate the amount of leachant to be added to the actual size of test portion and compensate for the moisture content in the test portion, as follows:

$$V_L = V - (\frac{100}{w_{dr}} - 1) \times M_d \tag{4}$$

where

V_L is the volume of leachant to be added, in ml;

V is the total volume of liquid in the test, in ml;

M_d is the dry mass of the test portion, in grams (g) (see 8.4);

w_{dr} is the dry residue of the waste, expressed as percentage (%) (see 8.3).

Prepare the leachant from demineralized water (see 6.1) and acid or base (see 6.2 or 6.3) according to the acid/base consumption for the relevant pH.

Prepare the acid adjusted leachant as follows:

$$V_L = V_{demin} + V_A \tag{5}$$

$$V_A = \frac{A \times M_d}{C_A} \tag{6}$$

where

V_L is the volume of prepared leachant, in ml;

V_{demin} is the volume of demineralized water used, in ml;

V_A is the volume of acid needed, in ml;

A is the acid consumption for the pH, in mol H^+/kg dry matter;

M_d is the dry mass of the test portion, in grams (g) (see 8.4);

C_A is the concentration of the acid, in mole per litre (mol/l) (see 6.2).

Prepare the base adjusted leachant as follows:

$$V_L = V_{demin} + V_B \quad (7)$$

$$V_B = \frac{B \times M_d}{C_B} \quad (8)$$

where

V_L is the volume of prepared leachant, in ml;

V_{demin} is the volume of demineralized water used, in ml;

V_B is the volume of base needed, in ml;

B is the base consumption for the pH, in mol OH^-/kg DM;

M_d is the dry mass of the test portion, in grams (g) (see 8.4);

C_B is the concentration of the base, in mole per litre (mol/l) (see 6.3).

Split the volume V_L of leachant into 3 equal fractions V_L/3.

9.3.3 Leaching procedure

Carry out the test at a temperature of 20 °C ± 5 °C.

Select the appropriate bottle size according to the test portion size. For M_d =15 g, 30 g and 60 g, this means respectively bottle sizes of 250 ml, 500 ml and 1 000 ml.

Clean the bottle before use by filling it with 1 mol/l nitric acid (see 6.2), leaving it for at least 24 h and then flushing it out with demineralized water (see 6.1).

Place one of the test portions in the rinsed bottle.

Add the leachant volume V_L/3 at three different times:

— 1st fraction at t_0;

— 2nd fraction at t_0 + 30 min;

— 3rd fraction at t_0 + 2 h.

Close the bottle and agitate the suspension (see 7.2.1) between each leachant addition. Measure and record pH before addition of fractions 2 and 3, and prepare additional bottles with modified acid/base addition, if deviations are observed from the expected pH at that time.

Continue to agitate after the last leachant addition until t = t_0 + 48 h.

For some waste materials gas formation may occur (e.g. CO_2, H_2). It is recommended to pay attention when opening the bottles to release the pressure (preferably in a fume hood). Also during the test visible pressure build-up may occur as reflected by swelling of the bottle. In such case, the pressure can be released during the test.

For the high pH values (pH > 9) CO_2 uptake may affect the leaching process. This may be reduced by minimizing the contact time with the air during handling.

Measure and record the pH at t_0 + 4 h, t_0 + 44 h, t_0 + 48 h. For the measurement of pH stop the agitation and allow the mix to settle for 5 min. Measure pH by inserting the electrode into the leachant in the bottle.

NOTE 1 The pH value at t_0 +4 h is used for checking that sufficient pH correction has been obtained by the acid or base additions.

Since the pH is measured directly in the suspension, rinse the pH electrode thoroughly and dry before and between uses in order not to contaminate the suspension.

The pH value measured before filtration at t_0 + 48 h will be the one associated to the analysis of the eluate. Measure conductivity (7.2.7) and optionally redox potential (7.2.8).

NOTE 2 pH is measured directly in the bottle at t_0 + 48 h since filtration can change pH in the eluate.

Report the pH deviation between t_0 + 4 h and t_0 + 44 h.

The deviation between pH at t_0 + 44 h and pH at t_0 + 48 h shall not exceed 0,3 pH units, which is the limit for approaching equilibrium (see 3.3). A special note shall be made in the report if this requirement is not met.

If too many experimental pH points deviate (more than 3 amongst 8), the conclusion is that the material has extreme sensitivity to pH in specific pH domains, which will be of importance in subsequent interpretation of the test results and shall be specifically noted in the test report.

NOTE 3 When approaching equilibrium is necessary for specific use but not fulfilled in the test, it is possible to continue the test to maximum 7 d for all the 8 experimental pH points (selected pH values) in order to avoid association of results at 48 h with those at longer leaching time. In this case, pH deviation could be limited to 0,3 pH units for the last 24 h (maximum between the 6^{th} and 7^{th} days).

The experimental pH point(s), which do not conform to these new conditions, will not be exploited and this fact shall be mentioned in the report. This specific procedure is not part of this European Standard.

Allow the suspended solids to settle for 15 min ± 5 min.

Rinse the part of filtration device, which is in contact with the eluate with nitric acid (see 6.2), and flush with water (6.1) before any use.

Filter the suspension through a 0,45 µm membrane filter (see 7.2.5) using the filtration device.

Proceed immediately with the eluate treatment as specified in Clause 10.

9.4 Natural pH

Repeat 9.3.3 for a test portion but without acid or base addition. Measure of pH after 4 h, 44 h and 48 h as in 9.3.3.

The pH value measured before filtration at t_0 + 48 h will be the one associated to the analysis of the eluate. Measure conductivity (7.2.7) and optionally redox potential (7.2.8).

A very low conductivity in the eluate at natural pH may increase generation of colloids, which may clog filters and increase analyzed concentrations. This can be avoided by addition of $NaNO_3$ in the leachant at a concentration comparable to the concentration of acid or base in the closest pH values tested.

The use of $NaNO_3$ (amount and concentration) shall be reported.

10 Eluate treatment, storage and analysis

Divide the eluate into an appropriate number of sub-samples for different chemical analysis. Preserve the eluate sub-samples depending on the elements to be analyzed and store them, in accordance with the requirements in EN ISO 5667-3.

Determine the concentrations of constituents of interest using the methods of eluate analysis according to EN 16192.

11 Blank test

The contribution of the leachant, devices and membrane filters to the result shall be determined by running a blank test periodically in order to check how the whole procedure performs.

The frequency of blank testing is related to the number of tests a laboratory performs and shall be assured in the quality control system of the laboratory.

Obtain different blank tests by carrying out the procedure according to 9.3 and 10 without the solid material with the addition of:

— both the maximum amounts of acid and base to the leachant of similar volume as used in the tests;

— acid used to obtain pH 2;

— base used to obtain pH 12.

The eluates of the blank tests shall fulfil the minimum requirement that the concentration of each considered element shall be less than 10 % of the concentration in the eluate. If the concentration of an element is below the detection limit for the analytical method applied, the blank test requirements will be fulfilled if the blank test is below the same limit of detection. If this requirement is not fulfilled it is necessary to reduce contamination.

The blank test results shall be available.

Do not subtract the results of the blank test determination from the test results.

NOTE The test is aiming at approaching equilibrium in which the contribution from the equipment is included. Subtraction of the blank test results will therefore lead to an incorrect equilibrium concentration.

12 Performance characteristics

The performance characteristics of the method as determined in a parallel inter-comparison validation between comparable US EPA method 1313 and this European Standard (EN 14429) are given in Annex E. Table 1 gives the resulting typical values for repeatability and reproducibility limits as their observed ranges. The typical value is derived from the data in Tables E.2 and E.3 by taking the median value and rounding the numbers.

Table 1 — Typical values and observed ranges of the repeatability and reproducibility limits

Results of the validation of the pH dependent leaching behaviour of substances from waste and stabilized waste	Typical value %	Observed range %
Repeatability limit, *r*	10	*4 - 25*
Reproducibility limit, *R*	26	*6 - 61*
The reproducibility limit provides a determination of the differences (positive and negative) that can be found (with a 95 % statistical confidence) between a single test result obtained by a laboratory using its own facilities and another test result obtained by another laboratory using its own facilities, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard and the two laboratory samples are obtained from the same primary field sample and prepared under identical procedures. The repeatability limit refers to measurements obtained from the same laboratory, all other conditions being identical. The reproducibility limit and the repeatability limit do not cover sampling but cover all activities carried out on the laboratory sample including its preparation from the primary field sample.		

NOTE The repeatability limit (*r*) and the reproducibility limit (*R*) as given in Tables E.2 and E.3 and in Table 1 are indicative values of the attainable precision, if the determination of the pH dependent leaching behaviour of substances from waste and stabilized waste is performed in accordance with this European Standard [EN 14429].

A limited number of materials and parameters were tested. Consequently, for other materials and parameters, performance characteristics may fall outside the limits as derived from the validation of the determination of the pH dependent leaching behaviour of substances from waste and stabilized waste. In particular for relatively heterogeneous materials, the repeatability and the reproducibility limits may be larger than the values given in Tables E.2 and E.3 and in Table 1.

13 Test report

In order to conform to this European Standard include the following information in the test report:

a) reference to this European Standard;

b) date of receipt of the laboratory sample;

c) sampling report according to 8.1;

d) date of the test (beginning and end);

e) complete identification of the laboratory sample (including dry residue);

f) pretreatment (e.g. method of size-reduction, drying, sub-division) and storage conditions;

g) temperature range during the performance of the test;

h) selected pH interval and number of test portions tested (N) within this interval;

i) dry mass of the test portion (g);

j) acid volumes (ml) and concentrations (mol/l), base volumes (ml) and concentrations (mol/l) and the corresponding amounts of H^+ or OH^- (mol/kg DM);

k) leachant volume (ml) and the resulting L/S (l/kg);

l) gas formation, when appropriate;

m) pH values at $t_0 + 4$ h, $t_0 + 44$ h and $t_0 + 48$ h, and difference in pH between $t_0 + 4$ h and $t_0 + 44$ h and between $t_0 + 44$ h and $t_0 + 48$ h;

n) the method of preservation for the different elements in accordance with Clause 9;

o) the analytical report in accordance with Clause 9;

p) the date of the latest blank test. Blank results shall be available upon request;

q) any deviation from the test method and the reason of this deviation together with all circumstances that have influenced the results;

r) record the data for the test results on a data sheet.

NOTE An example of the data sheet is given in Annex A.

Annex A
(informative)

Example of a data sheet

Table A.1 — An example of a data sheet for the recording of test results

	Unit	**Bottle no.**							
		1	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Dry mass of the test portion	g								
Acid volume	ml								
Acid concentration	mol/l								
Base volume	ml								
Base concentration	mol/l								
H^+ or OH^-	mol/kg DM								
Leachant volume	ml								
Resulting L/S	l/kg								
pH at t_0 + 4 h									
pH at t_0 + 44 h									
pH at t_0 + 48 h									
ΔpH (t_0 + 4 h) - (t_0 + 44 h)									
ΔpH (t_0 + 44 h) - (t_0 + 48 h)									
Constituent 1 :	mg/l								
Constituent 2 :	mg/l								
Constituent 3 :	mg/l								
Constituent 4 :	mg/l								
Constituent 5 :	mg/l								
Constituent 6 :	mg/l								
etc.									

Annex B
(informative)

Operation and uses of the test — Influence of pH on the leaching behaviour

B.1 Clarification of the two modes of operation of the test: influence of pH on the leaching behaviour

Both EN 14429 and EN 14997 are aiming at determining the influence of pH on the release of the inorganic constituents from a waste material.

In the test described in this European Standard (EN 14429), an equilibrium condition is established at different pH values as a result of the reaction between pre-selected amounts of acid or base and test portions of the waste material. Size reduction is performed to facilitate approaching of equilibrium.

In addition to the pH influence on leaching, the test addressing the influence of pH on leaching by continuous pH control is suitable for solubility control at a precisely specified pH. The test addressing the influence of pH on leaching by continuous pH control can be particularly suitable when materials are tested which have a very low buffer capacity or in the case of measurement of pH influence on leaching at a pH where a small pH change leads to strong change in release.

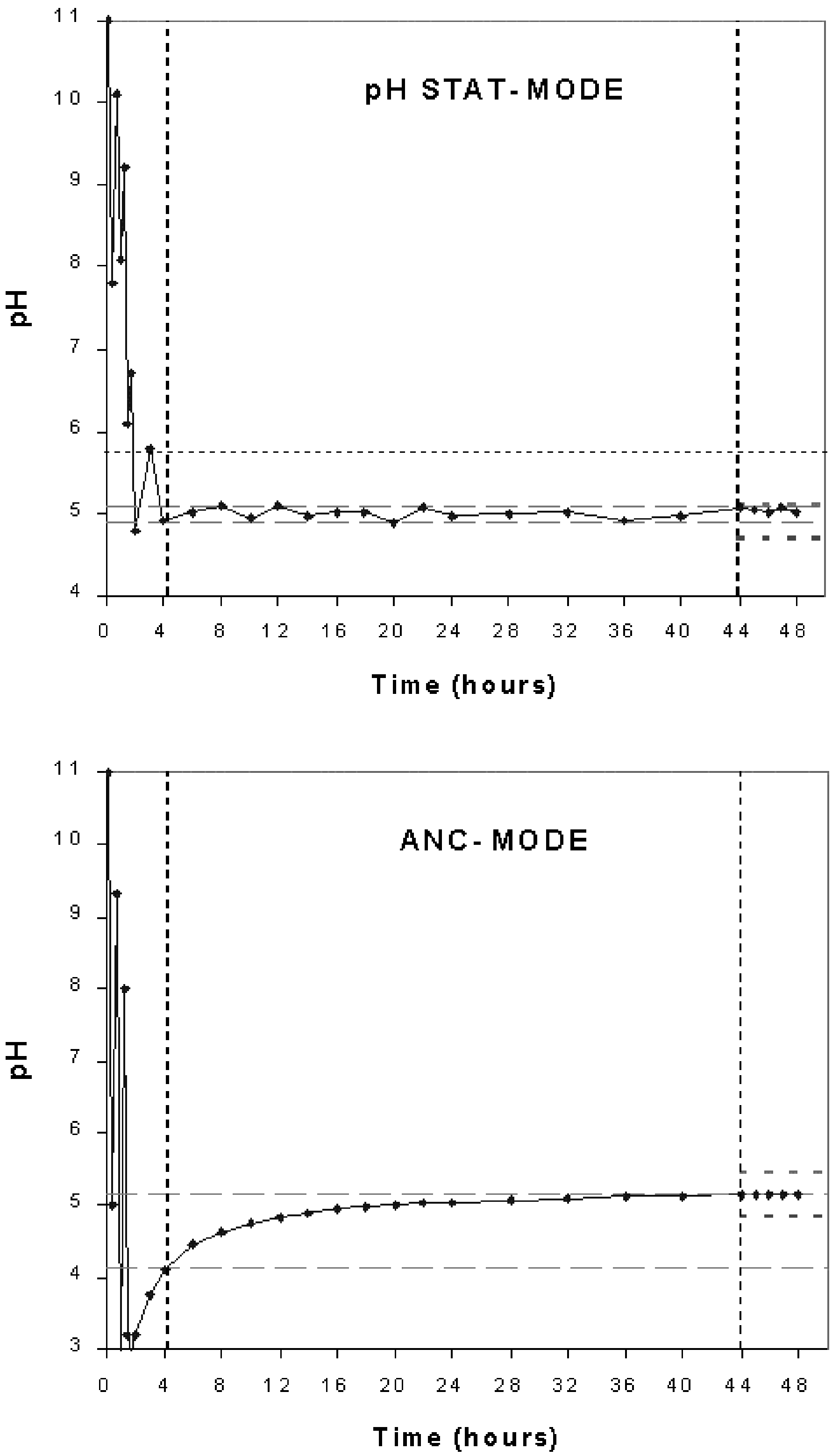


Figure B.1 — Typical pH variations during the two modes of the tests for determining the influence of pH on the leaching behaviour of an alkaline material at a final pH around 5

B.2 Expression of results

Three graphical presentations of the results can be obtained. They provide a visual representation of the test results as a trend (see B.3):

1) pH at t_0 + 48 h (see 9.3.2) versus the amounts of acid/base added (ANC and BNC curve) expressed in mol H^+/OH^- added per kg of dry matter;

2) concentration of each analyzed constituent in eluates in log scale in mg/l versus pH at t_0 + 48 h;

3) concentration of each analyzed constituent in eluates in linear scale in mg/l versus the amounts of acid or base added (mmol of H^+/OH^-/kg dry matter).

NOTE In the third visual representation, pH at t_0 + 48 h can also be added as a second ordinate.

In case when the leached amounts (U_X) (mg/kg dry waste material) are needed, they can be directly calculated by multiplying the concentrations in mg/l by the *L*/*S* value (normally *L*/*S* =10 l/kg dry material).

B.3 Scope and limits of the application field of the test

B.3.1 Remarks on the use of pH dependence test results to assess the effects of ageing

Leaching behaviour depends on pH, and the pH of a material may change in practice. For alkaline materials, the pH changes mainly as a result of carbonation. Carbonation is a natural process by which CO_2 from the air is taken up by alkaline materials. As a result, carbonation of alkaline materials is an important factor resulting in pH changes and changes in mineral composition of the material (mainly formation of carbonate minerals). Ageing and weathering are more generic terms which may include several simultaneous changes in a material (e.g. carbonation and oxidation/reduction processes) and can lead to formation of more stable minerals or incorporation of substances in more stable sorptive phases.

From experimental work on fresh alkaline materials and the same material in carbonated form in the laboratory or from the field (e.g. cementitious products, various slag types, municipal solid waste incinerator bottom ash), the main change in leachability is observed for Ca (calcite formation), Mg, Ba and Sr as a result of carbonation processes. In many cases trace elements are exhibiting similar leaching characteristics as in fresh products, which may be explained by other solubility controlling factors than those affected by carbonation [16, 17, 18, 19]. As such, testing a fresh product will provide valuable information on how leaching would change as a function of pH upon carbonation. The nature of this information is only qualitative as long as only the fresh product is tested, because trace elements such as metals are known to show a different pH dependent leaching behaviour as a result of carbonation reactions as compared to fresh material [21, 22]. In addition, the characterization of the fresh material does not necessarily address chemical changes appropriate when substances like Ba, Ca, Mg, and Sr are of interest (a characterization of a carbonated material would give a better estimate in that case).Therefore, testing both fresh and aged material (e.g., after a pretreatment) will elucidate significant changes in element leachability more quantitatively as a result of such changes that are also relevant under field conditions [16, 17, 18, 19, 21, 22].

For test interpretation purposes, it is important to realize that in case of bulk applications of alkaline materials, the exposed outer boundaries of the material are affected by contact with the atmospheric (CO_2 and O_2), soil atmosphere (about 3 times higher CO_2 level) and neutralization by soil buffer capacity. Although the interior may maintain alkaline conditions for a long time, the outer exposed surface determines to a large extent the observed impact (depending on the conditions of use). Depending on the pH dependent behaviour of a given element, this may result in lower release (e.g. Pb) or increased release (e.g. oxyanions) compared to the material properties of the freshly produced product.

Another use of the pH dependence test results can be to elucidate the chemical mechanisms that are controlling the leaching of major and minor elements. To enable such an assessment, a range of pH values in the test is necessary that is partly outside the window that is relevant for neutral or alkaline construction products as it is in practice (e.g., pH 8 - 12). This is expressed in the range of pH values that should be covered in the test, that extends from pH 2 to pH 12 (9.2). Knowledge of these mechanisms is relevant for possible changes in leaching over longer time frames and/or to create a basis for improvement of the environmental quality. For that purpose, a combination of testing and geochemical modelling can be used where hypothesis on the leaching mechanisms can be assumed and tested by comparison with the observed leaching behaviour from the pH dependence test. Examples of these applications are given in references [10, 12, 16, 18, 21 and 22].

The pH dependence test is suitable for a wide range of hazardous, non-hazardous waste, municipal solid wastes, industrial slags, mining wastes, a wide range of construction products, soils, contaminated soil, sediments, sludge, vitrified waste, stabilized waste, air dust, and low level nuclear waste. The method is fit for all inorganic substances as typically 30 major, minor and trace elements are analyzed by ICP-OES and additional methods for anions (e.g. Cl, Br, F, sulphate). In addition, the method has been applied to radionuclides (Tc-99, Pb-210, Po-210). And finally, the method has been applied for organic contaminants, which not themselves but through their interaction with DOC (dissolved organic carbon) are affected by pH changes [20].

B.3.2 Examples of test result use and considerations on scope

This test provides information on the influence of pH on leaching under the experimental conditions specified in this European Standard [5 - 10]. It does not directly take the effects of other parameters such as influence of other acids and bases than the nitric acid/sodium hydroxide used in the test, dissolved organic carbon, complexation, redox conditions into account.

This test method is a parameter specific test as specified in EN 12920. The application of this test method alone is not sufficient for the determination of the detailed leaching behaviour of a waste under specific conditions.

NOTE This generally requires the application of several test methods, behavioural modelling and model validation as specified in EN 12920.

Therefore, provided that the nitric acid/sodium hydroxide used in the test as well as the other experimental conditions are relevant for the considered scenario, this test is useful to:

— qualify and quantify the material resistance to acid-basic attack through the concentration measured, as a function of pH and the acid/base amount needed to reach a given final pH;

— identify the chemical behaviour trends and the availability levels of components at different pH values under the experimental conditions specified in this test. These values can be used as input to modelling of chemical behaviour using geochemical speciation models (e.g. MINTEQA2, GEOCHEM WORKBENCH, PHREEQC, ECOSAT, ORCHESTRA, CHESS, SPEC, etc.). In many cases, it also provides insight in the relevance of particular solubility controls and release mechanisms (e.g. formulate hypothesis on the dissolution mechanisms);

— provide a basis of reference for different leaching tests, as it has been shown that pH is one of the major controlling factors distinguishing tests from one another;

— compare leaching behaviour, with respect to pH, of the same parameter from different wastes or different material classes to be able to demonstrate similarities in solubility controlling conditions irrespective of material matrix;

— provide data to feed dynamic behavioural models, for instance under the following relationship: solubilization = f (pH or meq H^+/g) in the physico-chemical context linked with the presence of the other compounds in the material [11, 12]. This is not always possible with available literature data.

On the contrary, this test is not meant for:

— quantifying a maximum removable fraction as the concentrations obtained correspond to steady state situation close to chemical equilibrium. For example, the values obtained for the lowest pH and at high pH can only be considered as approaching the maximum removable fraction of respectively metals and oxyanions; reaching a plateau in release may indicate such a maximum;

— simulating actual situations in specific scenarios, as in addition at least information on low L/S will be needed.

B.4 Example: Identification of the sensitivity of leaching to pH over the environmentally relevant pH range

The test provides insight in the sensitivity of leaching of components from a specific material to pH (see Figure B.2). This factor has been found to be a major release controlling parameter in virtually all materials. Obviously, the relevant pH range for a given application may be limited. However, for characterization purposes the full pH range from at least 2 to 12 is important as different uses of the information relate to different pH domains. In Figure B.2 the leaching behaviour under the influence of pH is illustrated for Cd from heavily sewage sludge amended soil [5]. An indication of the repeatability of the method can be obtained from the duplicate test data. The test was performed with initial acid/base addition. This Figure B.2 indicates also pH ranges typical of some waste – conditions of scenarios combinations.

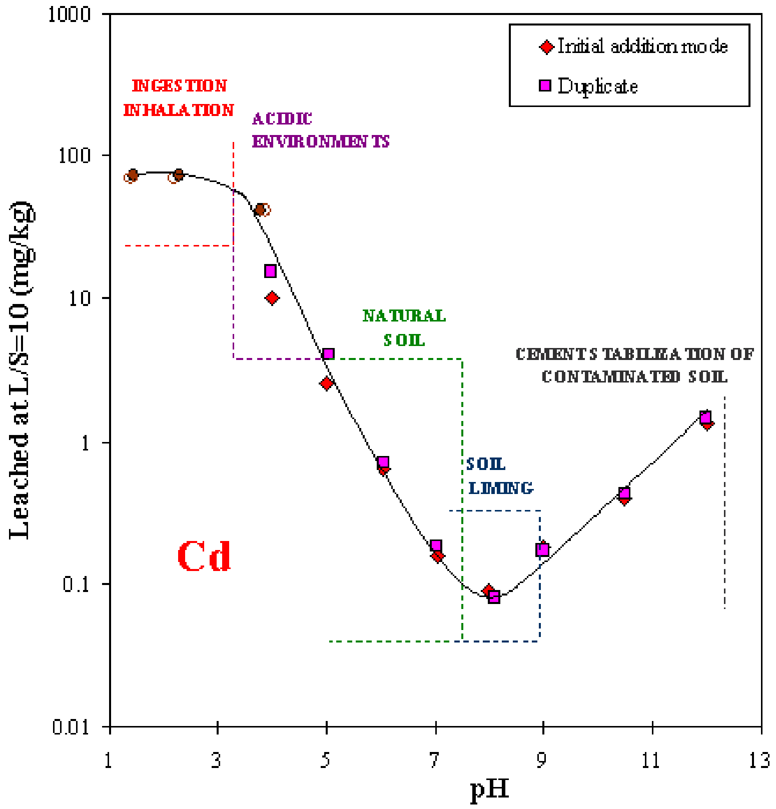


Figure B.2 — Illustration of the influence of pH on the leaching behaviour of a heavily sewage sludge amended soil as obtained in a pH range 2 - 12 (test performed with initial acid/base addition) its use in relation to different scenarios for the same material

Annex C
(informative)

Preliminary determination of the acid/base consumption

C.1 General

In order to determine the amount and concentration of acid/base two methods are possible:

1) a titration procedure to estimate the ANC and the BNC;

2) an arbitrary division of the maximum acid/base consumption for the extreme pH values.

C.2 Titration procedure to estimate the ANC and the BNC

C.2.1 Reagents and laboratory devices

a) Bottles made of polypropylene (PP), PTFE or polyethylene (PE).

b) Stirring or agitation device: this is a magnetic stirring device, using a polytetrafluorethylene (PTFE) coated magnetic stirring rod, or a mechanical stirring device, made of glass or PTFE.

c) Nitric acid, $c(HNO_3)$ = 0,1 mol/l to 14,4 mol/l.

d) Sodium hydroxide, c(NaOH) = 0,1 mol/l to 5 mol/l.

e) Distilled water, demineralized water or water of equivalent purity (5 < pH < 7) with a conductivity < 0,5 mS/m.

f) pH meter, with an accuracy of at least 0,05 pH units.

f) Titrator (optional).

C.2.2 Test portion

Test portions are prepared in accordance with the procedure in Clause 8. Based on sample heterogeneity, it is recommended that the test portion size is either M_d = 15 g, 30 g or 60 g (dry weight) (with a tolerance of ± 10 %).

C.2.3 Procedure

C.2.3.1 Preparation

Place the test portions in rinsed bottles, one for acid titration and the other for alkaline titration. The test aims at a final *L*/*S* ratio of 10 after acid or base addition. If the *L*/*S* exceeds 11 because of the high acid or base consumption of the material at the specific pH value a stronger acid or base should be used for pH adjustment.

Add an amount V of demineralized water in the bottles establishing a liquid to solid ratio (*L*/*S*) about 9. Calculate the volume V as follows assuming the density of water to be 1 g/ml:

$$V = 9 \times M_{\text{d}} - \left(M_{\text{w}} - M_{\text{d}}\right) \qquad \text{(C.1)}$$

where

- V is the amount of demineralized water added in the bottles, in ml;
- M_{w} is the undried mass of the test portion, in grams (g);
- M_{d} is the dried mass of the test portion, in grams (g);

Record the amount *V* of water added.

NOTE If information is available on the material concerning a particularly strong ANC or BNC another initial *L*/*S* can be used to allow the final *L*/*S* to remain ≤ 11.

C.2.3.2 Natural pH

Put the two filled bottles on the agitation device. Agitate or stir for 1 h. Determine the pH of the eluate directly in the bottles after settling for 10 min.

C.2.3.3 Acid titration

Add a portion of acid (C.2.1 c)), manually or by use of the titrator, into one of the bottles from C.2.3.1 and determine the pH directly in the bottle after 30 min agitation or stirring. Record the amount and concentration of acid added, and the pH obtained.

The amount of acid needed to get a decrease in pH varies between materials and the initial portions therefore need to be small in order to see the magnitude of the first response by the material.

In case of high acid demand manual addition of strong acid in the beginning of the determination is practical and a shorter response time than 30 min may be used.

Continue to add portions of acid and to measure pH after 30 min stirring or agitation after each portion added. Repeat until the entire pH interval from the natural pH (C.2.3.2) to pH 2 or below is obtained and the distance between the obtained pH values is smaller than 1,5 pH values. Record the amounts and concentrations of acid added, and the pH values obtained.

C.2.3.4 Base addition

Add a portion of base (C.2.1 d)) into the other of the bottles from C.2.3.2, and determine the pH directly in the bottle after 30 min agitation or stirring. Record the added amount and concentration of base, and the obtained pH.

The amount of base needed to get an increase in pH varies between materials and the initial portions therefore need to be small in order to see the magnitude of the first response by the material.

In case of high base demand manual addition of strong base in the beginning of the determination is practical and a shorter response time than 30 min may be used.

Continue to add portions of base and to measure pH after 30 min stirring or agitation after each portion added. Repeat until the entire pH interval from the initial pH (C.2.3.2) to pH 12 or above is obtained and the distance between the obtained pH values is smaller than 1,5 pH value.

In order not to under estimate the ANC or the BNC it is recommended to wait 24 h for pH 2 in case of very alkaline materials or pH 12 for materials with a high buffer capacity.

C.2.4 Expression of results

Make a graph of pH versus amounts of acid and base expressed in mol H^+/kg and mol OH^-/kg (for the graphical presentation express mol OH^-/kg as – mol H^+/kg).

C.3 Arbitrary division of the maximum acid/base consumption for the extreme pH values

C.3.1 General

For waste materials with a very strong acid-base capacity, manual titration may lead to excessive experimental duration when the maximum acid and base consumption in order to reach respectively pH 2 and pH 12 is unknown. In this procedure the natural pH and acid and base consumption at pH 2 and pH 12 respectively is estimated.

C.3.2 Reagents and laboratory devices

a) Bottles made of polypropylene (PP), PTFE or polyethylene (PE).

b) Stirring or agitation device: this is a magnetic stirring device, using a polytetrafluorethylene (PTFE) coated magnetic stirring rod, or a mechanical stirring device, made of glass or PTFE.

c) Titrator.

d) Nitric acid, $c(HNO_3)$ = 0,1 mol/l to 14,4 mol/l.

e) Sodium hydroxide, c(NaOH) = 0,1 mol/l to 5 mol/l.

f) Distilled water, demineralized water or water of equivalent purity (5 < pH < 7) with a conductivity < 0,5 mS/m.

C.3.3 Test portion

Test portions are prepared in accordance with the procedure in Clause 8. Based on sample heterogeneity, it is recommended that the test portion size is either M_d = 15 g, 30 g or 60 g (dry weight) (with a tolerance of ± 10 %).

C.3.4 Procedure

C.3.4.1 Preparation

Place two of the test portions in rinsed bottles, one for acid titration and the other for alkaline titration. The test aims at a final L/S ratio of 10 after acid or base addition. If the L/S exceeds 11 because of the high acid or base consumption of the material at the specific pH value a stronger acid or base should be used for pH adjustment.

Add an amount V of demineralized water in the bottles establishing a liquid to solid ratio (*L*/*S*) about 9. Calculate the volume V as follows assuming the density of water to be 1 g/ml:

$$V = 9 \times M_d - (M_w - M_d) \qquad \text{(C.2)}$$

where

V is the amount of demineralized water added in the bottles, in ml;

M_w is the undried mass of the test portion, in grams (g);

M_d is the dried mass of the test portion, in grams (g).

Record the amount V of water added.

NOTE If information is available on the material concerning a particularly strong ANC or BNC another initial LS can be used to allow the final LS to remain ≤ 11.

C.3.4.2 Natural pH

Put the two filled bottles on the agitation device. Agitate or stir for 1 h. Determine the pH of the eluate directly in the bottles after settling for 10 min.

C.3.4.3 Acid titration

Titrate one of the bottles in C.3.4.1 with acid (C.3.2 d)) in the titration equipment set at pH 2. Confirm the acid consumption over a titration period of 24 h. Record the added amount and concentration of acid, and the obtained pH.

If the time taken to complete this last pH point needs to be reduced, samples may be size reduced to below 0,5 mm (as no analysis is foreseen).

If this equipment is not available, manual titration may be carried out with the objective of achieving pH 2 as soon as possible including an overnight waiting period to validate the last measurement pH points.

C.3.4.4 Base addition

Titrate the other of the bottles in C.3.4.1 with base (C.3.2 e)) in the titration equipment set at pH 12. Confirm the base consumption over a titration period of 24 h. Record the added amount and concentration of base, and the obtained pH.

If this equipment is not available, manual titration may be carried out with the objective of achieving pH 12 as soon as possible including an overnight waiting period to validate the last measurement pH points.

If the time taken to complete this last pH point needs to be reduced, samples may be size reduced to below 0,5 mm (as no analysis is foreseen).

C.3.5 Expression of results

The acid consumption to reach pH 2 and the base consumption to reach pH 12 is recorded together with the natural pH.

Divide the amount of acid by double the number of pH values intended to be tested within the acid pH range (pH range as a result of acid addition).

Divide the amount of base by double the number of pH values intended to be tested within the alkaline pH range (pH range as a result of base addition).

EXAMPLE If e.g. between the natural pH and pH 2, 5 pH values are sought and if the maximum acid consumption is 5 mol H^+/kg, prepare 10 different acid solutions from 50 mmol H^+/l to 500 mmol H^+/l at evenly-spaced intervals.

NOTE 1 This method allows limitation of the misevaluation of the ANC and BNC and the selection of the 7 solutions to be analyzed after reaching the stationary pH. This allows also choosing solutions leading to the same pH, i.e. along a potential pH plateau corresponding to the buffer capacity of the material (e.g. carbonates) of high interest in term of behaviour.

NOTE 2 Due to buffering of the matrix equally spaced portions generally will not lead to proper final pH values. Doubling the number of bottles is no guarantee for obtaining properly spaced final pH values. Through interpolation, it will be possible to estimate the proper amounts needed from the curve of final pH against acid/base consumption.

Annex D
(informative)

Examples of acid and base neutralization capacities for waste, soil, sediment and construction materials

D.1 Examples of acid and base neutralization capacities

From previous work [5, 6, 7, 8, 9, 10] data on acid - and base neutralization capacity data have been obtained for a wide range of materials. This implies that this previous knowledge can be used in 9.3.2 of the pH dependence test. The information provided may not be entirely accurate for a sample under consideration. However, the variation within one material class is generally not very large.

In Figure D.1 acid neutralization and base neutralization capacities for waste, soil and construction materials are given, which can be used as starting point to carry out a pH dependence test. This information allows the selection of the relevant acid or base strength as well as the approximate amount of acid or base needed for a given sample weight to reach a certain desired end pH. This information can come in the place of the pre-titration as described in Annex C of the standard.

The selection of the acid strength is based on the buffer capacity of the material. In case of low buffer capacity in materials such as soil, metallurgical slag, sintered brick and sintered aggregates, the lowest acid/base strength specified in the standard should be used.

For the pH dependence test using continuous pH control, which is aimed at reaching a fixed and pre-determined pH, the acid strength is the most crucial parameter. For the pH dependence test with fixed initial acid/base addition, the proper amounts of acid/ base to be added needs to be determined. Generally more bottles are prepared in this latter test mode, from which the proper end pH values are selected for further analysis.

To calculate the amount of acid or base needed from the ANC / BNC expressed in mol/kg the following formula applies:

$$A_{\text{pHX}} = 1\ 000 \times ANC_{\text{pHX}} \times \frac{M}{N_{\text{pHX}}} \quad \text{(D.1)}$$

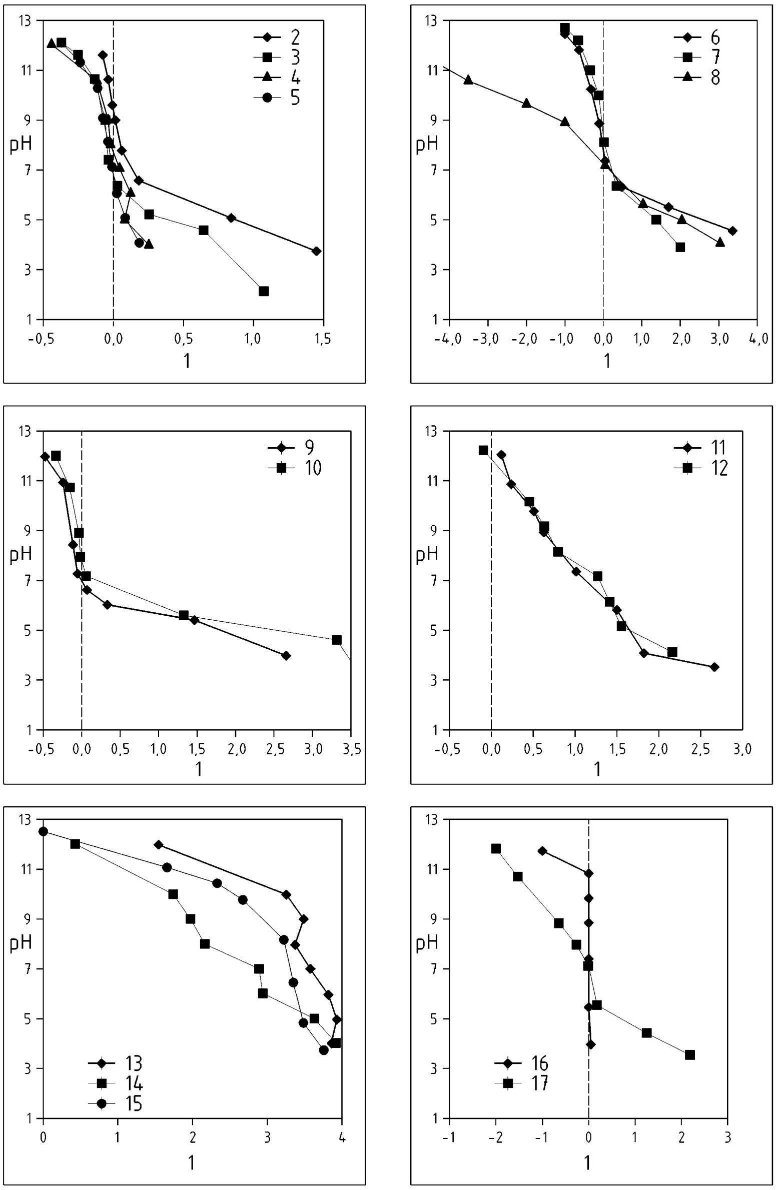
where

A_{pHX} is the amount of acid needed to reach pH = X for sample of weight M, in ml;

ANC_{pHX} is the acid neutralization capacity at pH = X, in mol/kg;

N_{pHX} is the acid normality selected based on the buffer capacity around pH = X, in mol/l;

M is the sample weight, in kg.



Key

1 ANC/BNC (mol/kg)

2 Contaminated soil – A

3 Contaminated soil – B

4 Eurosoil 4 (SMT4–CT96–2066)

5 Eurosoil 6 (SMT4–CT96–2066)

6 CW1 Compost from integral MSW

7 CW5 Compost from source separation

8 Sewage sludge (rural) – SEW1

9 Cont. river sediment – SED3

10 Lake sediment – SED1

11 MSWI Bottom ash

12 MSWI BA (SMT4-CT96-2066)

13 Fly ash cement – C1FA

14 Blast furnace slag cement – C2FA

15 Portland cement

16 Metallurgical slag

17 Ni - Sludge

NOTE The base addition is given as negative values.

Figure D.1 — Acid/base neutralization curves for a wide range of materials

D.2 Use of acid and base neutralization capacity data

These ANC/BNC characterization test data can be used to assess the amount of acid (as H^+) or base (as OH^-) needed to reach a given final pH in a waste. This property can be used also to determine the final pH of mixed waste in a landfill by summing the buffer capacities (provided that there is no chemical interaction between the different wastes). The data can also be used to assess the effect of external influences, such as carbonation and oxidation (acid production resulting from sulphide oxidation) on the pH of a waste. (as a first approximation, only considering the H^+ providing, without taking into account the specific effect of carbonation or sulphate in solution on the solubility of the different elements). It can be used also to assess the development of a pH front with potential consequences for changes in leaching behaviour or durability of cement-based materials (carbonation may lead to loss of structural integrity of cement stabilized waste). The external influences need to be calculated in the same units to allow comparison, so carbonation would have to be assessed through volume of gas, concentration of CO_2, conversion to corresponding moles of H^+ neutralizing capacity.

Annex E
(informative)

Repeatability and reproducibility data

E.1 Materials used in the interlaboratory comparison study

The interlaboratory comparison of the pH dependent leaching behaviour of substances from waste and stabilized waste was carried out with 10 American and 2 European laboratories on 3 materials. The materials selected for the interlaboratory comparison were chosen to represent waste and stabilized waste (detailed information can be found in the final report on the interlaboratory comparison study mentioned in [13,14]).

Table E.1 provides a list of the types of materials chosen for testing and the substances analyzed.

Table E.1 — Material types tested and substances analyzed in the interlaboratory comparison of the pH dependent leaching behaviour of substances from waste and stabilized waste

Grain size class	Sample code	Material type tested
Very fine material	EaFA	Coal fly ash
Fine grained (< 2 mm)	CFS	Contaminated soil from a smelter site
Size reduced monolithic material	SWA	Cement stabilized waste

E.2 Interlaboratory comparison results

The statistical evaluation was conducted according to ISO 5725-2 after log transformation of the test results. The average values, the relative repeatability standard deviation (RSD_r) and the relative reproducibility standard deviation (RSD_R) were obtained (Tables E.2 and E.3).

Table E.2 — Results of the interlaboratory comparison studies of the pH dependent leaching behaviour of substances from waste and stabilized waste (Part 1)

Concentration				**Concentration range**				**Labs**		**Collected**	**Filtered**	**Used**	**Concentration range**				
EPA Method	**Matrix**	**Parameter**	**pH**	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	EN	pH	Min mg/l	Max mg/l	RSD_r %
1313	CFS	Sb	2,0	0,99	1,76	8	17	8		27	0	27	14429	2,6	1,97	2,22	6
			4,0	0,64	1,22	13	19	8		27	0	27		4,4	0,923	1,01	5
			5,5	0,071	0,24	21	43	8		27	0	27		5,5	0,106	0,125	9
			7,0	0,022	0,051	18	23	8		27	0	27		6,8	0,026	0,038	19
			8,0	0,016	0,049	16	33	8		27	0	27		8,4	0,035	0,038	4
			9,0	0,024	0,044	17	21	8		27	0	27		9,6	0,043	0,046	3
			10,5	0,020	0,038	14	18	8		27	0	27		10,5	0,037	0,043	8
			12,0	0,005	0,020	18	47	8		27	0	27		12,3	0,010	0,013	12
			13,0	0,004	0,018	43	74	8		27	0	27					
1313	CFS	As	2,0	48,5	77,6	8	71	8		27	0	27	14429	2,6	20,6	69,9	52
			4,0	0,99	2,98	24	26	8		27	0	27		4,4	0,768	1,03	15
			5,5	0,071	0,27	29	37	8		27	0	27		5,5	0,121	0,138	6
			7,0	0,014	0,050	19	31	8		27	0	27		6,8	0,027	0,034	11
			8,0	0,013	0,047	25	37	8		27	0	27		8,4	0,036	0,053	21
			9,0	0,022	0,048	16	24	8		27	0	27		9,6	0,060	0,067	6
			10,5	0,054	0,136	9	25	8		27	0	27		10,5	0,082	0,091	6
			12,0	0,122	0,311	12	30	8		27	0	27		12,3	0,158	0,179	6
			13,0	0,107	0,705	29	85	8		27	0	27					

Concentration				Concentration range				Labs		Collected	Filtered	Used			Concentration range		
EPA Method	**Matrix**	**Parameter**	**pH**	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	EN	pH	Min mg/l	Max mg/l	RSD_r %
1313	CFS	Ba	2,0	0,004	0,010	15	136	8		27	0	27	14429	2,6	0,010	0,025	44
			4,0	0,022	0,041	13	19	8		27	0	27		4,4	0,028	0,029	3
			5,5	0,020	0,049	9	36	8		27	0	27		5,5	0,020	0,021	2
			7,0	0,059	0,129	7	36	8		27	0	27		6,8	0,095	0,099	2
			8,0	0,060	0,123	6	20	8		27	0	27		8,4	0,061	0,068	5
			9,0	0,064	0,107	4	14	8		27	0	27		9,6	0,067	0,074	5
			10,5	0,098	0,194	13	22	8		27	0	27		10,5	0,103	0,110	3
			12,0	0,006	0,018	27	35	8		27	0	27		12,3	0,007	0,025	68
			13,0	*0,006*	0,382	108	280	8		27	0	27					
1313	CFS	B	2,0	0,665	0,903	6	7	8		27	0	27	14429	2,6	0,89	1,20	17
			4,0	0,476	0,706	4	12	8		27	0	27		4,4	0,65	0,70	4
			5,5	0,270	0,381	8	9	8		27	0	27		5,5	0,35	0,35	1
			7,0	0,140	0,203	7	9	8		27	0	27		6,8	0,16	0,16	3
			8,0	0,100	0,156	8	10	8		27	0	27		8,4	0,24	0,25	1
			9,0	0,091	0,174	7	14	8		27	0	27		9,6	0,30	0,32	2
			10,5	0,123	0,173	5	9	8		27	0	27		10,5	0,45	0,47	2
			12,0	0,060	0,114	11	15	8		27	0	27		12,3	0,82	1,06	14
			13,0	0,050	0,104	12	24	8		27	0	27					

Concentration				**Concentration range**				**Labs**		**Collected**	**Filtered**	**Used**	**Concentration range**				
EPA Method	**Matrix**	**Parameter**	**pH**	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	EN	pH	Min mg/l	Max mg/l	RSD_r %
1313	CFS	Cd	2,0	41,9	51,4	4	11	8		27	0	27	14429	2,6	57,1	61,6	4
			4,0	32,4	52,5	6	15	8		27	0	27		4,4	51,8	54,1	2
			5,5	13,9	41,4	18	25	8		27	0	27		5,5	27,0	27,5	1
			7,0	1,9	8,6	28	63	8		27	0	27		6,8	4,3	4,5	3
			8,0	0,07	4,6	55	233	8		27	0	27		8,4	0,16	0,19	7
			9,0	0,012	0,25	35	132	8		27	0	27		9,6	0,013	0,013	2
			10,5	*0,001*	0,005	51	68	8		27	0	27		10,5	0,002	0,004	33
			12,0	*0,001*	0,010	49	114	8		27	0	27		12,3	*0,001*	*0,001*	NA
			13,0	0,002	0,4	108	158	8		27	0	27					
1313	CFS	Ca	2,0	1 379	1 720	5	7	8		27	0	27	14429	2,6	1 728	2 022	8
			4,0	1 109	1 427	4	7	8		27	0	27		4,4	1 370	1 385	1
			5,5	800	1 005	5	7	8		27	0	27		5,5	971	980	1
			7,0	628	737	3	5	8		27	0	27		6,8	703	728	2
			8,0	587	706	2	5	8		27	0	27		8,4	691	720	2
			9,0	563	712	2	6	8		27	0	27		9,6	671	680	1
			10,5	536	666	3	6	8		27	0	27		10,5	653	674	2
			12,0	526	670	3	7	8		27	0	27		12,3	686	696	1
			13,0	195	681	8	51	8		27	0	27					

Concentration				Concentration range				Labs		Collected	Filtered	Used	Concentration range				
EPA Method	Matrix	Parameter	pH	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	EN	pH	Min mg/l	Max mg/l	RSD_r %
1313	CFS	Pb	2,0	7,99	12,5	6	16	8		27	0	27	14429	2,6	8,3	10,4	12
			4,0	4,95	7,27	5	10	8		27	0	27		4,4	5,65	5,69	1
			5,5	4,06	7,49	12	19	8		27	0	27		5,5	7,56	7,67	1
			7,0	0,61	1,83	17	42	8		27	0	27		6,8	0,88	0,93	3
			8,0	0,11	1,2	26	112	8		27	0	27		8,4	0,07	0,12	26
			9,0	0,067	0,31	15	53	8		27	0	27		9,6	0,06	0,08	17
			10,5	0,13	1,1	45	80	8		27	0	27		10,5	0,08	0,20	42
			12,0	3,0	13	32	50	8		27	0	27		12,3	6,3	9,2	22
			13,0	0,007	13	152	350	8		27	0	27					
1313	CFS	Mo	2,0	0,070	0,27	22	47	8		27	0	27	14429	2,6	0,05	0,09	30
			4,0	0,004	0,027	43	47	8		27	0	27		4,4	0,015	0,016	5
			5,5	0,004	0,013	11	35	8		27	0	27		5,5	0,009	0,014	25
			7,0	0,008	0,018	17	21	8		27	0	27		6,8	0,016	0,017	4
			8,0	0,009	0,16	27	110	8		27	0	27		8,4	0,089	0,105	8
			9,0	0,049	0,46	21	114	8		27	0	27		9,6	0,44	0,47	4
			10,5	0,49	1,2	7	30	8		27	0	27		10,5	0,91	0,92	1
			12,0	1,20	1,89	4	15	8		27	0	27		12,3	1,27	1,38	4
			13,0	0,85	2,7	15	36	8		27	0	27					

Concentration				Concentration range				Labs		Collected	Filtered	Used	Concentration range				
EPA Method	Matrix	Parameter	pH	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	EN	pH	Min mg/l	Max mg/l	RSD_r %
1313	CFS	Se	2,0	0,41	0,86	9	30	8		27	0	27	14429	2,6	0,34	0,52	21
			4,0	0,21	0,29	6	11	8		27	0	27		4,4	0,28	0,29	2
			5,5	0,095	0,17	12	17	8		27	0	27		5,5	0,13	0,14	5
			7,0	0,059	0,11	14	16	8		27	0	27		6,8	0,10	0,13	15
			8,0	0,076	0,17	14	19	8		27	0	27		8,4	0,12	0,14	8
			9,0	0,097	0,18	9	19	8		27	0	27		9,6	0,18	0,20	4
			10,5	0,23	0,56	9	28	8		27	0	27		10,5	0,32	0,35	4
			12,0	0,31	0,68	12	21	8		27	0	27		12,3	0,79	0,94	9
			13,0	0,26	0,77	20	46	8		27	0	27					
1313	CFS	V	2,0	0,35	0,56	8	14	8		27	0	27	14429	2,6	0,40	0,59	21
			4,0	0,044	0,111	12	27	8		27	0	27		4,4	0,057	0,063	5
			5,5	0,028	0,053	4	20	8		27	0	27		5,5	0,042	0,044	2
			7,0	0,024	0,043	4	21	8		27	0	27		6,8	0,032	0,034	3
			8,0	0,024	0,042	3	20	8		27	0	27		8,4	0,031	0,034	5
			9,0	0,022	0,041	3	19	8		27	0	27		9,6	0,031	0,032	2
			10,5	0,020	0,040	3	25	8		27	0	27		10,5	0,029	0,030	1
			12,0	0,018	0,042	5	28	8		27	0	27		12,3	0,029	0,030	2
			13,0	0,013	0,049	7	54	8		27	0	27					

RSD_r is repeatability standard deviation

RSD_R is reproducibility standard deviation

p is the number of labs remaining in statistical evaluation

NOTE 1 All laboratories conducted 3 replicates of each test with one of the laboratories (the reference laboratory) conducting 6 replicates.

NOTE 2 No outlier analysis was performed.

NOTE 3 No data were removed from analysis (i.e., "filtered") due to blank concentrations.

NOTE 4 No data were "filtered for cause".

Concentration				**Concentration range**				**Labs**		**Collected**	**Filtered**	**Used**			**Concentration range**		
EPA Method	**Matrix**	**Parameter**	**pH**	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	EN	pH	Min mg/l	Max mg/l	RSD_r %

NOTE 5 Concentration data in italic indicates values set to 1/2 DLT.

NOTE 6 For the CEN test only RSD_r can be calculated.

Table E.3 — Results of the interlaboratory comparison studies of the pH dependent leaching behaviour of substances from waste and stabilized waste (Part 2)

	Parameter	EPA Method 1313	Concentration range				Labs		Collec-ted	Filtered	Used	EN 14429	Concentration range			EN 14997	Concentration range		
Matrix		**pH**	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	**pH**	Min mg/l	Max mg/l	RSD_r %	**pH**	Min mg/l	Max mg/l	RSD_r %
SWA	Sb	2,0	0,26	32	129	508	10	0	33	0	33	4,0	0,38	0,39	2	2,8	1,02	1,30	14
		4,0	0,26	1,8	26	74	10	0	33	0	33	5,1	0,35	0,48	16	4,5	0,21	0,33	28
		5,5	0,41	1,6	14	43	10	0	33	0	33	7,1	0,71	0,78	5	7,0	0,27	0,30	5
		7,0	0,55	2,0	13	40	10	0	33	0	33	7,8	0,88	1,09	11	8,5	0,30	0,33	5
		8,0	0,66	2,1	13	35	10	0	33	3	30	8,3	1,33	1,48	6	9,8	0,36	0,39	5
		9,0	0,76	2,3	8	31	10	0	33	0	33	9,8	0,73	0,80	5	11,0	0,45	0,86	43
		10,5	0,83	2,7	20	42	10	0	33	3	30	11,3	0,53	0,60	7	11,9	0,67	1,3	45
		12,0	0,87	4,9	32	73	10	0	33	0	33	12,3	1,2	1,7	17	12,8	1,37	1,69	12
		13,0	1,6	8,5	27	49	10	0	33	0	33								
SWA	As	2,0	4,7	20	59	75	10	0	33	0	33	4,0	3,78	3,90	2	2,8	0,45	0,51	7
		4,0	1,2	5,1	20	58	10	0	33	0	33	5,1	0,88	1,03	8	4,5	0,07	0,12	11
		5,5	0,15	1,0	35	59	10	0	33	0	33	7,1	0,07	0,07	3	7,0	0,21	0,22	4
		7,0	0,09	0,41	19	41	10	0	33	0	33	7,8	0,12	0,15	13	8,5	0,33	0,36	6
		8,0	0,09	0,37	20	47	10	0	33	3	30	8,3	0,18	0,18	1	9,8	0,21	0,22	3
		9,0	0,12	0,77	31	62	10	0	33	0	33	9,8	0,14	0,16	4	11,0	0,22	0,30	16
		10,5	0,13	1,2	22	124	10	0	33	3	30	11,3	0,31	0,32	2	11,9	0,57	1,15	36
		12,0	0,29	4,6	34	122	10	0	33	0	33	12,3	1,9	2,3	10	12,8	5,63	5,93	3
		13,0	4,1	17	22	51	10	0	33	0	33								

	Parameter	EPA Method 1313	Concentration range				Labs		Collec-ted	Filtered	Used	EN 14429	Concentration range			EN 14997	Concentration range		
Matrix		**pH**	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	pH	Min mg/l	Max mg/l	RSD_r %	pH	Min mg/l	Max mg/l	RSD_r %
SWA	Ba	2,0	0,25	1,18	65	91	10	0	33	0	33	4,0	0,80	0,83	2	2,8	1,15	1,19	2
		4,0	0,55	1,04	10	20	10	0	33	0	33	5,1	0,82	1,16	21	4,5	1,75	1,79	1
		5,5	0,76	2,03	11	26	10	0	33	0	33	7,1	1,4	1,9	17	7,0	1,74	1,86	3
		7,0	0,86	1,49	7	17	10	0	33	0	33	7,8	1,25	1,43	7	8,5	1,92	2,03	3
		8,0	0,75	1,34	7	16	10	0	33	3	30	8,3	1,13	1,30	7	9,8	1,03	1,16	6
		9,0	0,60	1,24	8	21	10	0	33	0	33	9,8	0,95	1,12	8	11,0	0,55	0,64	7
		10,5	0,42	1,4	11	38	10	0	33	3	30	11,3	0,58	0,62	3	11,9	0,28	0,34	11
		12,0	0,12	0,58	14	51	10	0	33	0	33	12,3	0,05	0,07	13	12,8	0,10	0,12	10
		13,0	0,01	0,6	106	136	10	0	33	0	33								
SWA	B	2,0	1,6	9,4	28	66	10	0	33	0	33	4,0	4,94	4,98	1	2,8	6,26	7,00	6
		4,0	3,6	7,5	6	26	10	0	33	0	33	5,1	3,64	4,02	5	4,5	8,76	11,0	13
		5,5	3,3	6,9	8	25	10	0	33	0	33	7,1	2,15	2,50	8	7,0	3,93	4,26	4
		7,0	2,6	4,3	10	16	10	0	33	0	33	7,8	1,74	1,90	4	8,5	1,68	2,05	11
		8,0	1,8	3,0	9	14	10	0	33	3	30	8,3	1,24	1,24	1	9,8	0,77	0,83	4
		9,0	1,35	2,0	7	9	10	0	33	0	33	9,8	0,71	0,75	3	11,0	0,3	0,5	42
		10,5	0,60	1,40	9	18	10	0	33	3	30	11,3	0,15	0,16	4	11,9	0,3	0,7	61
		12,0	0,07	1,16	16	110	10	0	33	0	33	12,3	0,25	0,33	13	12,8	1,3	1,9	24
		13,0	0,66	3,4	18	54	10	0	33	0	33								

	Parameter	EPA Method 1313	Concentration range				Labs		Collec-ted	Filtered	Used	EN 14429	Concentration range			EN 14997	Concentration range		
Matrix		**pH**	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	pH	Min mg/l	Max mg/l	RSD_r %	pH	Min mg/l	Max mg/l	RSD_r %
SWA	Ca	2,0	1 500	6 000	46	61	10	0	33	0	33	4,0	3 070	3 080	1	2,8	3 930	4 030	1
		4,0	3 400	8 200	7	23	10	0	33	0	33	5,1	4 700	5 100	4	4,5	7 700	8 300	4
		5,5	3 900	10 100	12	28	10	0	33	0	33	7,1	8 050	8 500	3	7,0	6 600	6 800	1
		7,0	4 000	9 400	5	23	10	0	33	0	33	7,8	7 390	7 780	3	8,5	4 800	5 400	6
		8,0	3 100	7 800	8	25	10	0	33	3	30	8,3	5 060	5 200	1	9,8	2 600	3 100	11
		9,0	2 300	6 300	18	29	10	0	33	0	33	9,8	2 160	2 200	1	11,0	530	660	13
		10,5	300	3 100	28	94	10	0	33	3	30	11,3	210	220	2	11,9	85	120	18
		12,0	13	190	36	115	10	0	33	0	33	12,3	3,8	5,3	16	12,8	6,5	9,5	23
		13,0	0,2	20	60	133	10	0	33	0	33								
SWA	Cr	2,0	6,2	24	67	89	10	0	33	0	33	4,0	12,4	12,6	1	2,8	6,1	6,6	4
		4,0	0,7	7,4	29	70	10	0	33	0	33	5,1	1,37	1,53	5	4,5	0,20	0,23	7
		5,5	0,006	0,5	74	277	10	0	33	0	33	7,1	*0,001*	*0,001*	NA	7,0	0,035	0,039	6
		7,0	*0,001*	0,05	99	198	10	0	33	0	33	7,8	0,009	0,011	8	8,5	0,011	0,015	18
		8,0	*0,001*	0,03	76	144	10	0	33	3	30	8,3	0,011	0,011	1	9,8	0,017	0,041	53
		9,0	0,002	0,05	66	108	10	0	33	0	33	9,8	0,010	0,013	15	11,0	0,12	0,19	29
		10,5	0,003	0,12	53	149	10	0	33	3	30	11,3	0,021	0,034	24	11,9	0,39	0,67	34
		12,0	0,02	1,2	42	227	10	0	33	0	33	12,3	0,45	0,57	13	12,8	1,8	2,4	18
		13,0	0,6	7,5	59	102	10	0	33	0	33								

Matrix	Parameter	EPA Method 1313	Concentration range				Labs		Collec-ted	Filtered	Used	EN 14429	Concentration range			EN 14997	Concentration range		
		pH	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	pH	Min mg/l	Max mg/l	RSD_r %	pH	Min mg/l	Max mg/l	RSD_r %
SWA	Co	2,0	0,08	0,33	38	58	10	0	33	0	33	4,0	0,166	0,167	1	2,8	0,170	0,180	3
		4,0	0,10	0,25	10	26	10	0	33	0	33	5,1	0,11	0,13	7	4,5	0,096	0,107	6
		5,5	0,056	0,23	16	43	10	0	33	0	33	7,1	0,020	0,037	32	7,0	0,004	0,005	18
		7,0	0,009	0,16	57	82	10	0	33	0	33	7,8	0,006	0,007	8	8,5	0,003	0,003	8
		8,0	0,003	0,03	41	68	10	0	33	3	30	8,3	0,003	0,003	1	9,8	0,002	0,003	31
		9,0	*0,001*	0,01	37	50	10	0	33	0	33	9,8	*0,001*	0,002	42	11,0	0,001	0,001	14
		10,5	*0,001*	0,01	16	59	10	0	33	3	30	11,3	*0,001*	*0,001*	NA	11,9	*0,001*	*0,001*	100
		12,0	*0,001*	0,01	7	8	10	0	33	0	33	12,3	*0,001*	*0,001*	NA	12,8	*0,001*	*0,001*	100
		13,0	*0,001*	0,01	18	19	10	0	33	0	33								
SWA	Mo	2,0	0,003	0,04	106	148	10	0	33	0	33	4,0	0,003	0,003	10	2,8	0,006	0,008	14
		4,0	0,005	0,02	23	68	10	0	33	0	33	5,1	0,009	0,013	18	4,5	0,01	0,01	8
		5,5	0,007	0,03	32	42	10	0	33	0	33	7,1	0,056	0,079	17	7,0	0,12	0,13	4
		7,0	0,020	0,10	36	58	10	0	33	0	33	7,8	0,123	0,135	5	8,5	0,18	0,18	2
		8,0	0,037	0,16	15	41	10	0	33	3	30	8,3	0,133	0,137	2	9,8	0,18	0,20	3
		9,0	0,067	0,18	11	28	10	0	33	0	33	9,8	0,138	0,149	4	11,0	0,17	0,18	2
		10,5	0,089	0,19	6	20	10	0	33	3	30	11,3	0,153	0,158	2	11,9	0,15	0,16	1
		12,0	0,094	0,21	5	19	10	0	33	0	33	12,3	0,047	0,057	10	12,8	0,13	0,13	2
		13,0	0,082	0,18	9	23	10	0	33	0	33								

Matrix	Parameter	EPA Method 1313 pH	Concentration range Min mg/l	Max mg/l	RSD_r %	RSD_R %	Labs p	Outliers	Collec-ted Total number	Filtered Number < DLT	Used Total used	EN 14429 pH	Concentration range Min mg/l	Max mg/l	RSD_r %	EN 14997 pH	Concentration range Min mg/l	Max mg/l	RSD_r %
SWA	Se	2,0	9,3	69	41	88	10	0	33	0	33	4,0	29,2	29,5	1	2,8	42,4	43,3	1
		4,0	40	99	7	25	10	0	33	0	33	5,1	48	60	12	4,5	89	99	6
		5,5	58	130	9	27	10	0	33	0	33	7,1	109	116	3	7,0	122	124	1
		7,0	77	150	5	19	10	0	33	0	33	7,8	113	120	3	8,5	119	128	4
		8,0	76	140	6	18	10	0	33	3	30	8,3	108	112	2	9,8	117	126	4
		9,0	72	130	10	19	10	0	33	0	33	9,8	98	102	2	11,0	108	111	1
		10,5	64	128	5	20	10	0	33	3	30	11,3	101	103	1	11,9	106	109	2
		12,0	62	130	5	19	10	0	33	0	33	12,3	33	44	15	12,8	116	117	1
		13,0	54	116	10	22	10	0	33	0	33								
SWA	V	2,0	0,23	1,6	94	175	10	0	33	0	33	4,0	0,32	0,33	2	2,8	0,34	0,35	2
		4,0	0,06	0,31	29	53	10	0	33	0	33	5,1	0,09	0,12	17	4,5	0,17	0,19	5
		5,5	0,08	0,23	10	37	10	0	33	0	33	7,1	0,18	0,20	3	7,0	0,20	0,20	1
		7,0	0,11	0,26	5	27	10	0	33	0	33	7,8	0,20	0,21	2	8,5	0,17	0,18	2
		8,0	0,12	0,24	6	25	10	0	33	3	30	8,3	0,18	0,20	5	9,8	0,14	0,15	4
		9,0	0,12	0,27	10	26	10	0	33	0	33	9,8	0,12	0,12	2	11,0	0,10	0,11	5
		10,5	0,09	0,26	8	42	10	0	33	3	30	11,3	0,09	0,10	1	11,9	0,13	0,18	17
		12,0	0,09	0,37	14	47	10	0	33	0	33	12,3	0,14	0,14	2	12,8	0,49	0,59	10
		13,0	0,32	0,81	17	26	10	0	33	0	33								

	Parameter	**EPA Method 1313**	**Concentration range**				**Labs**		**Collec-ted**	**Filtered**	**Used**	**EN 14429**	**Concentration range**			**EN 14997**	**Concentration range**		
Matrix		**pH**	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	pH	Min mg/l	Max mg/l	RSD_r %	pH	Min mg/l	Max mg/l	RSD_r %
EaFA	Sb	2,0	*0,004*	0,011	29	38	11		36	5	31	4,0	0,026	0,041	22	2,8	0,017	0,022	14
		4,0	*0,004*	0,049	67	67	11		36	16	20	5,1	0,064	0,10	23	4,5	0,046	0,053	7
		5,5	0,050	0,14	17	32	11		36	22	14	7,1	0,177	0,192	4	7,0	0,189	0,202	3
		7,0	0,092	0,24	14	27	11		36	4	32	7,8	0,17	0,20	9	8,5	0,19	0,23	11
		8,0	0,10	0,25	12	27	11		36	1	35	8,3	0,17	0,19	5	9,8	0,215	0,228	3
		9,0	0,098	0,29	11	30	11		36	0	36	9,8	0,20	0,225	6	11,0	0,187	0,200	4
		10,5	0,11	0,27	10	35	11		36	0	36	11,3	0,179	0,205	7	11,9	0,158	0,177	6
		12,0	0,11	0,23	12	18	11		36	15	21	12,3	0,173	0,177	1	12,8	0,13	0,16	8
		13,0	0,16	0,28	11	20	11		36	6	30								
EaFA	As	2,0	0,47	3,2	11	64	11		36	0	36	4,0	0,045	0,056	11	2,8	0,3	0,8	55
		4,0	0,06	0,20	11	37	11		36	17	19	5,1	0,071	0,073	1	4,5	0,079	0,097	11
		5,5	0,04	0,19	14	57	11		36	17	19	7,1	0,057	0,072	12	7,0	0,104	0,109	3
		7,0	0,04	0,19	15	50	11		36	17	19	7,8	0,085	0,112	14	8,5	0,25	0,36	20
		8,0	0,09	0,46	18	41	11		36	9	27	8,3	0,175	0,189	4	9,8	0,67	0,69	2
		9,0	0,24	0,92	14	40	11		36	0	36	9,8	0,72	1,0	17	11,0	0,95	1,01	3
		10,5	0,60	1,5	12	28	11		36	0	36	11,3	1,1	1,6	19	11,9	1,12	1,35	10
		12,0	0,74	2,7	9	43	11		36	0	36	12,3	1,6	1,8	8	12,8	1,3	1,8	18
		13,0	2,7	14	15	47	11		36	0	36								

Matrix	Parameter	EPA Method 1313	Concentration range				Labs		Collec-ted	Filtered	Used	EN 14429	Concentration range			EN 14997	Concentration range		
		pH	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	pH	Min mg/l	Max mg/l	RSD_r %	pH	Min mg/l	Max mg/l	RSD_r %
EaFA	Ba	2,0	0,74	1,15	7	9	11		36	3	33	4,0	0,229	0,240	2	2,8	0,64	0,81	15
		4,0	0,21	0,42	8	20	11		36	3	33	5,1	0,238	0,250	2	4,5	0,41	0,45	5
		5,5	0,22	0,37	4	14	11		36	3	33	7,1	0,224	0,243	4	7,0	0,362	0,366	1
		7,0	0,23	0,35	4	11	11		36	3	33	7,8	0,250	0,275	5	8,5	0,39	0,42	4
		8,0	0,26	0,38	8	11	11		36	3	33	8,3	0,249	0,268	4	9,8	0,60	0,67	6
		9,0	0,36	0,71	9	21	11		36	3	33	9,8	0,417	0,454	5	11,0	0,71	0,77	4
		10,5	0,5	1,3	21	28	11		36	3	33	11,3	0,354	0,402	6	11,9	0,783	0,799	1
		12,0	0,746	1,597	12	23	11		36	3	33	12,3	1,10	1,20	4	12,8	0,814	0,862	3
		13,0	0,168	2,411	41	68	11		36	3	33								
EaFA	B	2,0	2,65	3,24	3	6	11		36	0	36	4,0	3,54	3,60	1	2,8	3,02	3,26	4
		4,0	3,31	3,92	3	5	11		36	0	36	5,1	4,09	4,17	1	4,5	4,02	4,03	1
		5,5	3,9	5,0	4	6	11		36	0	36	7,1	4,20	4,42	3	7,0	4,41	4,48	1
		7,0	4,1	5,7	6	6	11		36	0	36	7,8	4,35	4,45	1	8,5	4,30	4,35	1
		8,0	3,8	6,7	9	9	11		36	0	36	8,3	4,17	4,26	1	9,8	4,49	4,55	1
		9,0	3,8	4,5	3	5	11		36	0	36	9,8	3,72	4,03	4	11,0	3,82	3,95	2
		10,5	3,6	4,7	3	7	11		36	0	36	11,3	3,79	4,19	5	11,9	3,19	3,42	4
		12,0	3,2	5,0	4	13	11		36	3	33	12,3	3,57	3,66	1	12,8	2,66	2,97	6
		13,0	2,2	5,2	5	27	11		36	3	33								

Matrix	Parameter	EPA Method 1313 pH	Concentration range Min mg/l	Max mg/l	RSD$_r$ %	RSD$_R$ %	Labs p	Outliers	Collected Total number	Filtered Number < DLT	Used Total used	EN 14429 pH	Concentration range Min mg/l	Max mg/l	RSD$_r$ %	EN 14997 pH	Concentration range Min mg/l	Max mg/l	RSD$_r$ %
EaFA	Ca	2,0	99	142	4	8	11		36	0	36	4,0	147	150	1	2,8	137	144	2
		4,0	133	153	2	4	11		36	0	36	5,1	150	154	1	4,5	154	156	1
		5,5	142	170	4	5	11		36	0	36	7,1	150	158	3	7,0	156	157	1
		7,0	142	199	5	6	11		36	0	36	7,8	154	155	1	8,5	137	140	1
		8,0	128	223	9	10	11		36	0	36	8,3	142	146	1	9,8	115	118	1
		9,0	110	138	3	6	11		36	0	36	9,8	105	114	5	11,0	59	66	6
		10,5	74	102	4	9	11		36	0	36	11,3	83,3	87,4	3	11,9	29	38	16
		12,0	32	60	10	18	11		36	0	36	12,3	37,2	37,7	1	12,8	15	23	26
		13,0	8,6	23	17	33	11		36	0	36								
EaFA	Cr	2,0	1,3	2,0	7	14	11		36	0	36	4,0	0,45	0,50	6	2,8	0,90	1,10	11
		4,0	0,05	0,6	34	100	11		36	1	35	5,1	0,002	0,003	17	4,5	0,043	0,097	56
		5,5	0,001	0,05	103	205	11		36	10	26	7,1	0,030	0,043	19	7,0	0,017	0,022	14
		7,0	0,001	0,16	89	155	11		36	1	35	7,8	0,043	0,052	10	8,5	0,086	0,12	21
		8,0	0,01	0,35	31	100	11		36	0	36	8,3	0,088	0,099	6	9,8	0,22	0,28	14
		9,0	0,09	0,41	13	41	11		36	0	36	9,8	0,22	0,30	15	11,0	0,33	0,39	10
		10,5	0,18	0,46	10	28	11		36	0	36	11,3	0,36	0,40	5	11,9	0,45	0,47	3
		12,0	0,24	0,48	9	22	11		36	0	36	12,3	0,46	0,50	5	12,8	0,51	0,61	9
		13,0	0,34	0,67	11	22	11		36	0	36								

Matrix	Parameter	EPA Method 1313	Concentration range				Labs		Collec-ted	Filtered	Used	EN 14429	Concentration range			EN 14997	Concentration range		
		pH	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	pH	Min mg/l	Max mg/l	RSD_r %	pH	Min mg/l	Max mg/l	RSD_r %
EaFA	Co	2,0	0,35	0,48	5	7	11		36	0	36	4,0	0,219	0,226	2	2,8	0,29	0,35	11
		4,0	0,20	0,26	6	8	11		36	0	36	5,1	0,221	0,227	1	4,5	0,202	0,214	3
		5,5	0,13	0,26	11	15	11		36	0	36	7,1	0,048	0,053	5	7,0	0,046	0,051	5
		7,0	0,01	0,16	39	85	11		36	0	36	7,8	0,024	0,025	2	8,5	*0,001*	0,002	42
		8,0	*0,001*	0,067	50	159	11		36	15	21	8,3	0,003	0,005	29	9,8	*0,001*	*0,001*	100
		9,0	*0,001*	0,004	38	51	11		36	7	29	9,8	*0,001*	*0,001*	NA	11,0	*0,001*	*0,001*	100
		10,5	*0,001*	0,003	16	24	11		36	2	34	11,3	*0,001*	*0,001*	NA	11,9	*0,001*	*0,001*	100
		12,0	*0,001*	*0,001*	NA	NA	11		36	0	36	12,3	*0,001*	*0,001*	NA	12,8	*0,001*	*0,001*	100
		13,0	*0,001*	*0,001*	NA	NA	11		36	0	36								
EaFA	Mo	2,0	0,19	0,53	7	32	11		36	0	36	4,0	0,08	0,09	1	2,8	0,14	0,23	28
		4,0	0,087	0,154	6	16	11		36	1	35	5,1	0,16	0,16	1	4,5	0,1	0,2	45
		5,5	0,2	0,9	22	44	11		36	0	36	7,1	2,92	3,07	3	7,0	2,30	2,49	4
		7,0	0,8	3,5	26	32	11		36	0	36	7,8	3,28	3,35	1	8,5	3,02	3,18	3
		8,0	2,3	4,9	12	14	11		36	0	36	8,3	3,42	3,50	1	9,8	3,40	3,48	1
		9,0	2,88	4,07	3	9	11		36	0	36	9,8	3,3	3,6	4	11,0	3,54	3,62	1
		10,5	3,15	4,46	4	8	11		36	0	36	11,3	3,62	3,71	1	11,9	3,64	3,69	1
		12,0	3,35	4,45	4	4	11		36	0	36	12,3	3,64	3,70	1	12,8	3,66	3,70	1
		13,0	3,19	4,62	4	8	11		36	0	36								

	Parameter	**EPA Method 1313**	**Concentration range**				**Labs**		**Collec-ted**	**Filtered**	**Used**	**EN 14429**	**Concentration range**			**EN 14997**	**Concentration range**		
Matrix		**pH**	Min mg/l	Max mg/l	RSD_r %	RSD_R %	p	Outliers	Total number	Number < DLT	Total used	pH	Min mg/l	Max mg/l	RSD_r %	pH	Min mg/l	Max mg/l	RSD_r %
EaFA	Se	2,0	0,20	1,0	10	52	11		36	0	36	4,0	0,07	0,14	39	2,8	0,25	0,41	32
		4,0	0,04	0,24	21	43	11		36	25	11	5,1	0,17	0,19	5	4,5	0,15	0,17	7
		5,5	0,17	0,47	13	32	11		36	7	29	7,1	0,94	1,02	4	7,0	0,92	0,98	4
		7,0	0,34	1,6	16	35	11		36	1	35	7,8	1,15	1,27	5	8,5	1,8	2,2	10
		8,0	0,7	2,6	15	32	11		36	0	36	8,3	1,84	1,91	2	9,8	4,15	4,26	1
		9,0	2,0	4,0	7	21	11		36	0	36	9,8	4,26	4,66	4	11,0	5,33	5,42	1
		10,5	4,0	5,9	4	8	11		36	0	36	11,3	5,54	5,68	1	11,9	5,79	6,00	2
		12,0	5,1	6,6	4	7	11		36	0	36	12,3	6,01	6,15	1	12,8	6,09	6,11	1
		13,0	5,3	8,4	4	9	11		36	0	36								
EaFA	V	2,0	2,9	6,6	12	27	11		36	0	36	4,0	0,08	0,09	4	2,8	0,5	1,1	46
		4,0	0,08	0,23	17	32	11		36	0	36	5,1	0,07	0,07	2	4,5	0,10	0,13	12
		5,5	0,07	0,15	10	26	11		36	0	36	7,1	0,16	0,17	4	7,0	0,19	0,20	2
		7,0	0,11	0,38	11	40	11		36	0	36	7,8	0,19	0,21	7	8,5	0,29	0,33	7
		8,0	0,13	0,61	15	43	11		36	0	36	8,3	0,23	0,24	2	9,8	0,99	1,05	3
		9,0	0,22	1,1	11	47	11		36	0	36	9,8	0,8	1,1	16	11,0	1,87	2,01	4
		10,5	0,69	2,8	12	40	11		36	0	36	11,3	2,4	2,7	6	11,9	2,7	3,3	11
		12,0	2,0	3,6	7	15	11		36	0	36	12,3	3,00	3,07	1	12,8	3,7	5,2	19
		13,0	3,7	5,9	7	13	11		36	0	36								

RSD_r is repeatability standard deviation

RSD_R is reproducibility standard deviation

p is the number of labs remaining in statistical evaluation

NOTE 1 All laboratories conducted 3 replicates of each test with one of the laboratories (the reference laboratory) conducting 6 replicates.

Matrix	Parameter	EPA Method 1313 pH	Concentration range Min mg/l	Max mg/l	RSD_r %	RSD_R %	Labs p	Outliers	Collec-ted Total number	Filtered Number < DLT	Used Total used	EN 14429 pH	Concentration range Min mg/l	Max mg/l	RSD_r %	EN 14997 pH	Concentration range Min mg/l	Max mg/l	RSD_r %

NOTE 2 No outlier analysis was performed.

NOTE 3 No data were removed from analysis (i.e., "filtered") due to blank concentrations.

NOTE 4 Some data were "filtered for cause" due to procedural issues (e.g., pH measurements not consistent with test target structure).

NOTE 5 Concentration data in italic indicates values set to 1/2 DLT.

NOTE 6 For the CEN tests only RSD_r can be calculated.

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