

# Extraction of soluble Phosphorus

## Introduction

This document is developed in the project 'Horizontal'. It is the result of desk study “ Extraction of soluble phosphorus” in the project and aims at the description of the determination of soluble phosphorus in soil, sludge, biowaste, treated biowaste and related waste. After discussion with all parties concerned in CEN and selection of a number of test methods described in this study will be developed further as an modular horizontal method and validated in the project 'Horizontal' .

Until now test methods determining properties of materials were often prepared in Technical Committees (TC's) working on specific products or specific sectors. In those test methods often steps as sampling, extraction, release or other processing, analyses, etc were included. In this approach it was necessary to develop, edit and validate similar procedural steps over and over again for each other product.. Consequently this resulted in a lot of duplicate work. To avoid such duplication of work for parts of a testing procedure often was referred to parts of test methods from other TC's. However following problems are often encountered while using references in this way: 1).The referenced parts are often not edited in a way that they could easily be referred to, 2). the referenced parts are often not validated for the other type of material and 3) the updates of such test standards on products might lead to inadequate references.

In the growing amount of product and sector oriented test methods it was recognised that many steps in test procedures are or could be used in test procedures for many products, materials and sectors. It was supposed that, by careful determination of these steps and selection of specific questions within these steps, elements of the test procedure could be described in a way that they can be used for all materials and products or for all materials and products with certain specifications. Based on this hypothesis a horizontal modular approach is being investigated and developed in the project 'Horizontal'. 'Horizontal' means that the methods can be used for a wide range of materials and products with certain properties. 'Modular' means that a test standard developed in this approach concerns a specific step in a test procedure and not the whole test procedure (from sampling to analyses) but only.

The use of modular horizontal standards implies the drawing of test schemes as well. Before executing a test on a certain material or product to determine certain characteristics it is necessary to draw up a protocol in which the adequate modules are selected and together form the basis for the test procedure.

This standard is a module, for determining soluble phosphorus in solid and liquid materials . This module concerns with the extraction of soluble phosphorus in soil, sludge, biowaste, treated biowaste and related waste .

The texts of the chapters 1-11 are normative; annexes are normative or informative, as stated in the top lines of the annexes.

## 1 Normative references

EN ISO 3696, Water for analytical laboratory use – Specification and test methods  
EN 13040 Soil improvers and growing media – Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory compact bulk density  
EN 13651 Soil improvers and growing media- Extraction of calciumchloride/DTPA (CAT) soluble nutrients  
ISO 11464 Soil quality – Pretreatment of samples for physico-chemical analysis  
ISO 11465 Soil quality - Determination of dry matter and water content on a mass basis – gravimetric method  
EN 12880 Characterisation of sludge – Determination of dry residue and Water content  
CEN/TC 292 WI 292030 Characterisation of waste – Preparation of test portions from the laboratory sample

## 2 Scope and working area

This European Standard specifies an extraction method for the routine determination of calcium chloride/DTPA (CAT) extractable phosphorus (soluble phosphorus) in soil, sludge, biowaste, treated biowaste and related wastes. The method is not applicable to liming materials and some other materials.

Dry and moist (fresh) samples can be extracted.

## 3 Principle

A sample is extracted with calcium chloride/DTPA at room temperature in an extraction ratio of 1+8 (solid+liquid). The extracted soluble P is determined by ICP-OES or by a colourmetric method.

## 4 Reagents

### 4.1 General

All reagents shall be of recognised analytical grade

### 4.2 Dihydrated calcium chloride, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$

### 4.3 Diethylenetriaminepentaacetic acid(DTPA)

4.4 Concentrated extracting solution  $\text{CaCl}_2$ /DTPA (CAT): dissolve 14,7 g  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  (4.2) and 7,88 g DTPA (4.3) in 800 ml hot water (approximately 80 °C) with stirring on a magnetic stirrer in a 1000 ml beaker until all solid is dissolved (within about two hours). Allow to cool to room temperature, transfer the solution to a 1000 ml flask and dilute to the volume with water. The solution is stable at room temperature for several weeks. Any precipitation that occurs will disappear with warming and stirring.

4.5 Extracting solution : Dilute the concentrated extracting solution (4.4) with water in the ratio of 1+9 (concentrated extracting solution+water). The final concentration of the extracting solution should be 0,01 mol/l  $\text{CaCl}_2$  and 0,002 mol/l DTPA. The pH of the extracting solution should be adjusted if necessary to be between 2,6 and 2,65.

4.6 Nitric acid,  $c(\text{HNO}_3) \approx 15 \text{ mol/l}$ ,  $\approx 1,42 \text{ g/ml}$

4.7 Nitric acid,  $c(\text{HNO}_3) \approx 0,5 \text{ mol/l}$ , dilute 35 ml nitric acid (4.6) to one litre with water

## 5 Apparatus

### 5.1 General

All glass and plastic vessels used in the procedure should be subject to an appropriate preparation procedure in order to keep the risk of contamination as low as possible. It is recommended that all vessels are cleaned by carefully immersing in warm nitric acid (4.7) for a time and then rinsed with water. The apparatus consists of usual laboratory apparatus, and in particular the following:

5.2 Plastic bottles or containers, sufficiently large with screw cap to accommodate the volume of the sample, extraction solution and at least 10 % of the volume air volume

5.3 Shaking machine, capable of holding the plastic bottles or containers (5.2) and maintaining the sample in suspension without damaging the structure of the sample. The use of a horizontal table shaker is recommended.

Over head shaker: 30-35 rotations/min

Horizontal shaker: 140 movements/min (VDLUF A 1997, VDLUF A 2002)

5.4 Filter paper, cellulose-based ashless types, with a medium pore size

Note 1: Centrifugation is an alternative separation method

5.5 Analytical balance with an accuracy of 10 mg

## **6 Quality control**

Certified or reference samples with known and unchangeable content of soluble P are used to control the extraction.

## **7 Pretreatment of test samples**

All samples shall be pretreated according to the special standard in the field of soil, sludge, biowaste, treated biowaste and related waste. The test samples have to be homogenous and of a defined grain size.

## **8 Working instructions**

Note 2: Homogeneity of the laboratory and the test sample has to be guaranteed.

### **8.1 Extraction**

Weigh a portion (test sample) of the dried or fresh (field fresh) material to be analysed into the plastic bottle or container. Add the extracting solution (4.5) in the ratio of 1+8 (solid+liquid), secure the cap and shake for 1 h in the shaking machine (5.3) under defined conditions.

Note 3: See to the special conditions when analysing soil improvers or growing media as described in the standard EN 13651.

### **8.2 Filtration**

Filter the suspension immediately through the filter paper (5.4) discarding at least the first 10 ml of the filtrate. In some cases paper filtration is too slow or even impossible. In such cases alternative procedures for obtaining a clear extraction solution like centrifugation or decantation of the supernatant solution are acceptable.

Note 4: The alternative separation technique has to be reported.

Note 5: The filtrate is stable for about three days if stored at a temperature of 0-5 °C. The filtrate can be stored for a longer time in a deep freezer at about -18 °C. Before Using this deep frozen solution, the thawed solution shall be thoroughly mixed .

### **8.3 Blank**

The reagent blank test shall be carried out in parallel with the extraction, by the same procedure as outlined in 8.1 and 8.2 using the same quantities of the extraction solution without the test sample.

### **8.4 Determination of soluble P**

The determination of the extracted amount of soluble P is done with ICP-OES ( ISO 11885) or follows the procedure according to Hoffmann (Hoffmann et al.).

## **9 Calculation and expression of results**

Calculate the content of soluble phosphorus in mg/kg dry matter or mg/l on the basis of dry matter or the determined compacted laboratory bulk density.

## **10 Validation and Precision**

For information see Annex A.

## 11 Test report

The test report is formulated according to the special requirements of accreditation following the regulations of EN 17025.

### Annex A (informativ)

Validation data EN 13651

#### Bark humus

Analyt	No. of participants	Content, average, mg/l	$s_r$ mg/l	$ws_r$ %	$s_R$ mg/l	$s_R$ %
P	20	290,0	14,59	5,03	108,99	37,6

#### Biowaste

Analyt	No. of participants	Content, average, mg/l	$s_r$ mg/l	$s_r$ %	$s_R$ mg/l	$s_R$ %
P	18	42,9	1,30	3,03	12,54	29,2

#### Clay containing peat (fertilized)

Analyt	No. of participants	Content, average, mg/l	$s_r$ mg/l	$s_r$ %	$s_R$ mg/l	$s_R$ %
P	19	35,2	1,19	3,38	4,62	13,1

#### Coarse peat (fertilized)

Analyt	No. of participants	Content, average, mg/l	$s_r$ mg/l	$s_r$ %	$s_R$ mg/l	$s_R$ %
P	21	92,9	9,41	10,1	25,12	27,0

#### Composted sludge

Analyt	No. of participants	Content, average, mg/l	$s_r$ mg/l	$s_r$ %	$s_R$ mg/l	$s_R$ %
P	19	132,6	3,85	2,90	52,11	39,3

#### Composted wood fibre

Analyt	No. of participants	Content, average, mg/l	$s_r$ mg/l	$s_r$ %	$s_R$ mg/l	$s_R$ %
P	20	55,2	1,62	2,93	11,20	20,3

In the present table the figures for  $s_r$  % and  $s_R$  % were calculated again using the data from the appendix of the existing standard EN 13651. The original data were taken with the factor 2,8.

## **Annex B** (informativ)

### Bibliography

- CEN/TC 292 WI 292030 Characterisation of waste – Preparation of test portions from the laboratory sample
- EN ISO 3696, Water for analytical laboratory use – Specification and test methods
- EN 13040 Soil improvers and growing media – Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory compact bulk density
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- ISO 11464 Soil quality – Pretreatment of samples for physico-chemical analysis
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- ISO 11885 Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectrsocopy
- VDLUFA, 1997, Methodenbuch Band I, Bodenuntersuchung, Methode A 13.1.1, VDLUFA Verlag, Bonn
- VDLUFA, 2002, Methodenbuch Band I, Bodenuntersuchung, Methode A 6.4.1, VDLUFA Verlag, Bonn