

Extraction of soluble Phosphorus in Soil, Sludge, Biowaste and Treated Biowaste

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SUMMARY

The phosphorus chemical bindings are very different in the different materials as soil, sludge, biowaste and treated biowaste. The amount of P in the extraction solution of the different extraction methods depends on the extraction power of the respective applied methods. The necessity of harmonisation of the extraction methods for “soluble P” becomes clearly obvious for example in the paper written by Neyrod (Neyrod et al.). In the present paper a proposal to formulate the definition of “soluble P” is made. According to this definition the existing standards are critically discussed and assessed. Criteria which have to be fulfilled by the proposed standard are formulated. The assessment of the existing methods leads to the conclusion that the extraction following the procedure described in the standard EN 13651 (CAT-method) would be a possibility to describe the “soluble P” as fraction of total P in the materials soil, sludge, biowaste and treated biowaste.

The ruggedness test and the validation for all materials have to be done.

1. INTRODUCTION

Within the framework of the project “Horizontal” and the harmonisation of methods the discussion in the Steering Committee leads to the conclusion that there is a necessity of having an additional method for “soluble phosphorus” in this stage besides the determination of total phosphorus and the leaching methods. The following study deals with the comparison of the existing standards to analyse soil, sludge, biowaste and treated biowaste. In this study the behaviour of phosphorus in the environment is the main question. Eutrophication has become one of the major water pollution problems all over Europe. Phosphorus is regarded as a key factor responsible for eutrophication. The concentration of phosphorus in lakes and rivers results both from external inputs and internal loading from the sediment. Its release depends on the form of phosphate in the sediment. Not all forms of phosphorus are bioavailable and therefore, are likely to increase eutrophication. Dissolved P can be considered to contribute only a few percent to total P.

The knowledge of the amount of “soluble P” is an additional information in the process of the assessment of a material.

Methods to analyse different matrices for the purpose of fertilisation exist as separate methods for this special purpose and it is not the aim to replace these methods. Most of them are no international standards. As a consequence of that there are wide differences between the results from method to method and even within one method used by different laboratories (Neyrod et al.). There are a lot of different interpretation schemes based on long time field trials to calculate the fertilizer recommendation. Furthermore there are methods to quantify total phosphorus and different phosphorus forms in different matrices.

The question to answer first is: what is the definition of “soluble P”?

2. THE ENVIRONMENTAL BEHAVIOUR OF PHOSPHORUS

Within the different materials phosphorus exists in different forms of chemical bindings. It can be bound organically or in different forms of inorganic and/or adsorptive bindings. Besides the quantum of phosphorus which is bound in living organisms phosphorus occurs in the following conditions in soil (Boeckman et.al.):

- phosphate dissolved in soil solution
- unstable adherent to soil particles especially clay
- fixed phosphate in soil minerals and precipitated inorganic chemical bindings
- organic phosphorus bindings

The transformation of one form to another is reversible and runs with different time factors and in different processes (Hedley et al.). The phosphate concentration in soil solution is low (ca. 0,03 – 0,4 mg/l , Boeckman et al.). This results in 2kg soluble phosphate per hectare in the maximum. The situation is comparable for the other materials. Because of its endeavour to bind to the mineral component parts of soil, sludge or sediment or being built in the clay structures the danger of shifting and localised flattening by solved phosphate from the soil can be considered as low. One tries to describe the releasability of phosphorus by respective methods (leaching). The biggest part of phosphate entry into the surface water is caused by erosion. The shifting to deeper areas is mainly not caused by solved but by particle bound phosphate via the coarse pore system and clefts. The distribution of the percentage amounts of the different P-fractions may change when sludge, compost or other materials are disposed (Leineweber). In general, the amounts of P lost varies between the different pathways , the reported data show a range of

yearly loss > 20 kg/ha for erosion, data for surface run off are mostly between 1 and 5 kg/ha, and for leaching < 1 kg/ha (Chardon et al, Ruban et al)

The transformation of the form of binding of phosphorus is highly determined by the pH of the respective examined matrix. Besides, other properties such as the redox potential, kinetic of the ad- and desorption and others play a role of course. At all these processes an equilibrium sets between the different phases. Here the part of the dissolved phosphorus plays an inferior role while the liquid layer is very important for the intermediate step of the different chemical processes (Schernewski et al.). This situation can be transferred from soil to other materials, respectively.

Anyway, to answer the question concerning “soluble phosphorus” one has to chose a method that seems to be suitable for the materials that shall be examined. Here it has to be stressed that special methods which are used for the examination of materials for exactly defined problems such as e.g. agricultural fertilisation are -at least currently- not touched by the standardisation. They keep their validity for their special purpose.

Another criterion to propose one method as a draft standard is the possibility of acceptance by the different groups of experts in the different European countries. That means an already standardized method should be used if possible. This is also the task for this desk study.

The further discussion of the significance of phosphorus fractions refers to desk study 16 (Janssen) or to desk study 23 (v.d.Sloot et al.) for questions of extractability, resp. releasability.

3. EXISTING STANDARDS OR DRAFT STANDARDS

Table 1 gives a survey over the existing standards which have to be considered when discussing the question of soluble phosphorus.

Table 1 Existing standards or draft standards for determination of soluble Phosphorus

Standard Nb.	S=soil SL=sludge W=waste BO= biowaste and treated biowaste, soil improvers	validation data available	liquid/solid ratio	extractant pH	year of publication
ISO 11263	S	No	20/1	0,5 mol/l Na ₂ CO ₃ 8,5	1994
EN 13651	BO	Yes	5/1	0,01mol/l CaCl ₂ , 0,002 mol/l DTPA 2,65	2002
EN 13652	BO	Yes	5/1	Water 7,0	2002
ISO/DIS 21268-2	S	No	10/1	0,001 mol/l CaCl ₂	2004
ISO/DIS 21268-3	S	No	10/1	0,001 mol/l CaCl ₂	2004
PrCEN 14429	W	No	10/1	pH-adjusted acid or base	2004
EN 12457-1-4	W	Yes	the ratio differs	Water	2002

3.1 ISO 11263 Soil quality – determination of phosphorus – Spectrometric determination of phosphorus soluble in sodium hydrogen carbonate

This international standard specifies an extraction method and analytical conditions to determine the content of soil phosphorus soluble in sodium hydrogen carbonate solution. After the extraction step, two different methods of colour development are possible. This international standard is applicable to all types of soils. A soil, pretreated according to ISO 11464, is treated with a 0,5 mol/l sodium hydrogen carbonate solution at pH 8,5 in a ratio (solid/liquid) of 1:20 to reduce the concentration of calcium, aluminium and iron(III) ions by precipitation of calcium carbonate and aluminium and iron(III) hydroxides and release phosphate ions in solution. The clear extract is analysed for phosphorus by a spectrometric method involving the formation of an antimony-phosphate-molybdate complex (at room temperature) or a phosphate molybdate complex at high temperature both reduced with ascorbic acid to form a deep-coloured, blue complex.

This standard is used in several European countries to investigate the availability of P.

3.2 EN 13651 Soil improvers and growing media – Extraction of calcium chloride/DTPA (CAT) soluble nutrients

This European Standard describes an extraction method for the determination of nutrients and elements extractable with a calciumchloride/DTPA solution in soil improvers and growing media. This standard is not suitable for calcium and magnesium soil improvers and some other materials. The test sample is extracted at room temperature with a solution of 0,01 mol/l calciumchloride and 0,002 mol/l DTPA in a ratio of (solid/liquid) 1:5 . The nutrients and elements are determined by suitable determination methods.

This standard is used in several European countries to investigate the availability and to calculate the fertilisation demand of P in secondary raw materials, treated biowaste, soil improvers and growing media. In some countries the results of the determination of micro nutrients ins soils are used to calculate the additional demand of plants of these elements.

3.3 EN 13652 Soil improvers and growing media - Extraction of water soluble nutrients and elements

This European Standard describes an extraction method for the determination of nutrients and elements extractable with water in soil improvers and growing media. This standard is not suitable for calcium and magnesium soil improvers and some other materials. The test sample is extracted at room temperature with water in a ratio of (solid/liquid) 1:5. This method gives an attempt to approximate the phosphorus concentration in the liquid layer of the material under investigation. The nutrients and elements are determined by suitable determination methods.

3.4 ISO/DIS 21268 – 2 Soil quality - Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials - Part 2: Batch test using a liquid to solid ratio of 10 l/kg dry matter.

This International Standard specifies a test providing information on leaching of soil and soil materials under experimental conditions specified hereafter, and particularly at a liquid to solid ratio of 10 l/kg dry matter. It applies to soil and soil material, which has a particle size less or equal to 4mm. This International Standard has been developed to investigate the release of inorganic and organic constituents from soil and soil material and their ecotoxicological effects of eluates with respect to microorganisms, fauna and flora.

The test procedure specified in this International Standard produces an eluate, which is subsequently characterised by chemical existing determination methods. This test cannot be used alone to determine the total leaching behaviour of a soil. More leaching tests are needed for that extended goal.

3.5 ISO/DIS 21268- 3 Soil quality - Leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials - Part 3: Up flow percolation test

This International Standard specifies a test, which is aimed at determining the leaching behaviour of inorganic and organic constituents from soil and soil material. The method is a once-through percolation test with water (0,001 mol/l CaCl₂) under standardised conditions of flow rate. The material is leached under hydraulically dynamic conditions. The eluates obtained can be used to determine the ecological properties of the soil with respect to micro-organisms, flora and fauna. The test results enable the distinction between different release patterns, for instance wash-out and release under the influence of interaction with the matrix, when approaching local equilibrium between material and leachant.

This test method produces eluates, which can subsequently be characterised by physical, chemical and ecotoxicological methods according to existing standard methods. The results of eluate analysis are presented as a function of the liquid/solid ratio.

3.6 PrCEN/TS 14429:2004 Characterisation of waste – Leaching behaviour tests – Influence of pH on leaching with initial acid/base addition

This Technical Specification has been developed primarily to support the requirements for leaching behaviour testing within EU and EFTA countries. Tests to characterize the behaviour of waste materials can generally be divided into three categories. The relationship between these tests are summarized below:

1. "Basic characterization" tests are used to obtain information on the short and long term leaching behaviour and characteristic properties of waste materials. Liquid/solid ratios, leachant composition, factors controlling leachability such as pH, redox potential, complexing capacity, ageing of waste and physical parameters are addressed in these tests.
2. "Compliance" tests are used to determine whether the waste complies with a specific behaviour or with specific reference values. The tests focus on key variables and leaching behaviour previously identified by basic characterization tests.

The test procedure described in this Technical Specification belongs to category 1.

In the test, which is described in this Technical Specification 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 accelerate reaching of equilibrium.

This test is different from the "pH dependence test with continuous pH control" (pH static test see WI 00292033) 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.

This Technical Specification is applicable to determine the influence of pH on the leachability of inorganic constituents from a waste material. 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 chemically.

This Technical Specification is a parameter specific test as specified in ENV 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.

3.7 EN 12457 – 1: 2002 Characterisation of waste – Leaching - Compliance test for leaching of granular waste materials and sludges – Part 1: one stage batch test at a liquid to solid ratio of 2 l/kg for materials with high solid content and with particle size below 4 mm (with or without size reduction)

This part of four European Standards specifies a compliance test providing information on leaching of granular wastes and sludges under the experimental conditions specified in the standard, and particularly a liquid to solid ratio of 2 l/kg dry matter. It applies to waste which has a particle size below 4 mm without or with size reduction. The test procedure produces an eluate which shall subsequently be characterised by appropriate determination methods. The procedure is only applicable to waste material and sludges having a high solid content: the dry matter content ratio shall be higher than 33% . The minimum dry matter content ratio shall be high enough to allow a sufficient mixing of the leachant and the test portion.

3.8 EN 12457 – 2: 2002 Characterisation of waste – Leaching - Compliance test for leaching of granular waste materials and sludges – Part 2: one stage batch test at a liquid to solid ratio of 10 l/kg for materials with particle size below 4 mm (with or without size reduction)

This part of four European Standards specifies a compliance test providing information on leaching of granular wastes and sludges under the experimental conditions specified in the standard, and particularly a liquid to solid ratio of 10 l/kg dry matter. It applies to waste which has a particle size below 4 mm without or with size reduction. The test procedure produces an eluate which shall subsequently be characterised by appropriate determination methods.

3.9 EN 12457 – 3: 2002 Characterisation of waste – Leaching - Compliance test for leaching of granular waste materials and sludges – Part 3: Two stage batch test at a liquid to solid ratio of 2 l/kg and 8 l/kg for materials with high solid content and with particle size below 4 mm (with or without size reduction)

This part of four European Standards specifies a compliance test providing information on leaching of granular wastes and sludges under the experimental conditions specified in the standard, and particularly a liquid to solid ratio of 2 l/kg dry matter in a first step and subsequently of 8 l/kg dry matter in a second step. It applies to waste which has a particle size below 4 mm without or with size reduction

This procedure is only applicable to waste material and sludges having a high solid content : the dry matter content ratio shall be higher than 33%. In addition, the necessary quantity of eluate in each step shall be obtained to perform the chemical characterisation of the eluate. Furthermore, the minimum dry matter content ratio shall be high enough to allow a sufficient mixing of the leachant and the test portion.

3.10 EN 12457 – 4:2002 Characterisation of waste – Leaching - Compliance test for leaching of granular waste materials and sludges – Part 4: one stage batch test at a liquid to solid ratio of 10 l/kg for materials with particle size below 10 mm (with or without size reduction)

This part of four European Standards specifies a compliance test providing information on leaching of granular wastes and sludges under the experimental conditions specified in the standard, and particularly a liquid to solid ratio of 10 l/kg dry matter. It applies to waste which has a particle size below 10 mm without or with size reduction. The test procedure produces an eluate which shall subsequently be characterised by appropriate determination methods.

The procedure is only applicable to waste material and sludges having a high solid content: the dry matter content ratio shall be at least higher than 33% . In addition, the necessary quantity of eluate in each step shall be obtained to perform the chemical characterisation of the eluate. Furthermore, the minimum dry matter content ratio shall be high enough to allow a sufficient mixing of the leachant and the test portion.

3.11 Extraction methods in different European countries

There are a lot of other methods in several European countries to estimate the solubility, the availability and releasability of phosphorus. The results yielded with some of these extraction

methods are used to estimate the phosphorus availability and to estimate the additional phosphorus demand for plant nutrition. There exist different concepts to do this estimations which are based on field and pot trials of plant nutrition and fertilisation. (Neyrod et al.) . Several methods are listed in table 2, which are used in different European countries.

Table 2 Examples of used methods in different European countries to determine a phosphorus fraction in soil (and some other materials)

Country	Extraction method
Denmark (Andersen)	Sodium bicarbonate; CAT
France (Ciesielski)	Sodium hydrogen carbonate; monohydric citric acid; ammonium oxalat
Netherland (Chardon)	Water; calcium chloride; Mehlich II
Germany	DL; CAL; aqua regia; CAT
Austria (Danneberg)	DL; CAL; aqua regia; water
Poland (Fotyma et al.)	DL; 0,5 mol HCl
Tchech republic (Stana)	Mehlich III
Italy (Ciesielski)	Sodium bicarbonate; Bray-1
Hungary (Loch et al.)	Mehlich II
Slovakia (Filkorn et al.)	Ammoniumlactate/acetic acid
Belgium (P.Ehlert)	Mehlich II; EDTA-Ac
Finland (P.Ehlert)	Morgan
Greece (P.Ehlert)	Bray-1; sodium bicarboonate
Irland (P.Ehlert)	Morgan
Norway (P.Ehlert)	Mehlich II
Romania (P.Ehlert)	Mehlich II
Spain (P.Ehlert)	Bray-1; sodium bicarbonate
Sweden (P.Ehlert)	Mehlich II
United Kingdom (P.Ehlert)	Sodium bicarbonate

The amount of extracted P decreases from aqua regia to calcium chloride and water. The different methods are used for different purposes as described in the respective papers. Most of the described methods are used to calculate the demand of fertilizer for plant nutrition.

4. EVALUATION OF DRAFTING A HORIZONTAL STANDARD

4.1 Definition of soluble phosphorus and requirements and criteria for proposing a draft standard

In this desk study “soluble phosphorus” means phosphorus that is either present in the actual matrix in the actual liquid layer or that is dissolved by the respective method. Here the long lasting processes of desorption, solution and adjustment of equilibrium as they are respected in the “leaching standards” shall not be used for the examination of “soluble phosphorus”. These methods are summarized as “leaching methods”. The easily soluble phosphorus which passes into the extraction solution in short extraction resp. contact times shall be captured here. The adjustment of equilibria and fundamental change of the chemical structure of the phosphorus binding material shall not be done by the proposed method for this very question. In a certain way the method shall reflect the mobility of phosphorus, while this phosphorus fraction is certainly defined by the method itself. Here it has to be taken into consideration that in the frame of Horizontal not only soil but also sludge, biowaste and treated biowaste shall be examined. Consequently prescribed procedures such as time of shaking and speed of rotation of the shaker and the ratio of solid to liquid have to be defined exactly for the application of the method at a later time to configure the quite short process of the extraction in a reproducible way. The extracted amount of P depends on this procedures and the chosen method determines the quota of “soluble P” (Koopmans et al.).

Furthermore a contribution to the description of the examined material shall be made with the analysed “soluble P” as a fraction of the phosphorus pool (main goal). As a second goal the described method should also allow the determination of other elements next to phosphorus, for example trace elements and other nutrients and ions. This demand is made because the analytical work of the laboratories has to be simplified and rationalised.

The respective method should not be a method which records the different phosphorus fractions including total phosphorus gradually by sequential extraction procedures as described for example by Ruban et al. (Ruban et al. 2002). The method should record the soluble phosphorus with the defined steps of proceeding of this method in one operation.

The extraction should lead to a phosphorus concentration in the extraction solution that guarantees an unproblematic measurement under routine conditions. For this reason weak extraction solutions such as water or calciumchloride solutions (i.e. ISO 14255), ammoniumnitrate (DIN 17930) or others must not be taken into consideration. The laboratory risk of having contamination while handling material of very low analyte concentration has to be avoided.

The method should fulfil the requirements contributing to the total assessment of the phosphorus problematic of a special material.

In the matrices which are treated in this desk study the phosphorus appears in very different binding forms. For soil it is referred to chapter 2. In sludge phosphorus will mainly be bound to iron or aluminium and partly to Ca. That means it is not as easily releasable and extractable. For sewage sludge the percentage of the single fractions is especially dependent on pretreatment (precipitation and stabilisation) of the sludge. In natural sludges and sediments the situation is comparable. A further P-quota in sludge is also organically bound. In biowaste and treated biowaste phosphorus is mainly organically bound which means it is only slowly transformable, releasable and extractable. De Groot and Golterman have shown that organic P is partly composed of phytates (de Groot et al.).

In other waste materials the origin and genesis have to be taken into consideration. See also desk study 16, chapter 1.2.4 (Janssen).

Taking the different initial situations concerning the binding form of phosphorus and its chemical surrounding into consideration some experts have a disposition to abstain from the proposal of a uniform method for “soluble phosphorus” (Deller, Suntheim), saying that these results were of too low validity.

The most intensive work concerning the question of solubility of phosphorus and plant-availability was done in the past in connection with fertilizer recommendation and plant nutrition. This work was done from an agricultural perspective. Here only the matrix soil has been object of examination. Only recently other materials came into focus, too. For soil a variety of extraction methods is being applied (see table). They also aim on different phosphorus fractions but every one just describes a certain part which is normally defined by the method. Basing on these methods and on respective plant nutrition and fertilisation experiments extensive regional interpretation schemes for fertilisation recommendations have been developed. The proposed method cannot and shall not replace the basic extraction methods for these schemes since it is still lacking the respective plant nutrition and fertilisation experiments in the member states.

4.2 Evaluation of existing standards

The standards EN 12457,1-4 (Characterisation of Waste) and also the series ISO 21268, 2 -3 (soil quality) do not fulfil the requirements made above. In these standards there is the demand to extract the sample as long as the equilibrium of element under analysis is reached between the solid and the liquid layer up to 24 h. These methods serve for the observation of the leaching behaviour of materials and for the scrutiny of declaration of materials. This is not the purpose of a method for the observation of the proportion of “soluble phosphorus”. Consequently these standards are not suitable for a method for the determination of soluble phosphorus.

The standard ISO 11263 extracts with a solution of sodium hydrogen carbonate at a pH of 8.5 and exclusively attuned to the element phosphorus. The ratio of solid:liquid is 1:20. In some countries it is being used for the calculation of the additional demand of fertilizer for plant nutrition. Because of its chemistry this method heavily interferes into the binding form of phosphorus. In calcareous, alkaline or neutral soils (materials) calcium is precipitated as carbonate consequently the concentration of P is increased in the solution. The extractant rises the pH which causes the precipitation of iron and aluminium as hydroxides. Secondary precipitation reactions in acid and calcereous soils (materials) are reduced to a minimum because the concentration of aluminium, iron and calcium remains at a very low level in this extractant.

Especially for the coextraction of trace elements this method is not applicable because of the high pH.

The Standard EN 13652 extracts with water with a ratio solid:liquid of 1:5. The method is especially used for biowaste and treated biowaste for the extraction of elements including nitrate and ammonium. Aqueous extraction methods with other ratios of solid to liquid exist also for soils and waste. This method records only easily soluble phosphorus and gives no hint to possible availability of P from the pool as well.

This extraction creates problems especially with P because of the very low concentration especially with the extraction of soils in the extraction solution. There are severe problems with contamination during the analytical process. Therefore this method is not suitable as an extraction method for “soluble P” as a routine method.

The only method which fulfils the criteria and the definition of “soluble P” formulated above is the EN 13651, extraction of calcium chloride/DTPA (CAT) soluble nutrients. This method is

used in several countries and may be accepted as an extraction method to determine “soluble phosphorus”, nutrients and trace elements as well as ammonium and nitrate in one extraction procedure. (VDLUFA 1997,VDLUFA 2002). But with this method problems with the extraction of calcareous materials or materials with a higher pH can occur. For this question further research is required.

This method has to demonstrate its ruggedness not only for soil improvers and growing media (biowaste and treated biowaste) but also for soil and sludge. In some countries it is used to determine several constituents in soils, biowaste and treated biowaste .

In the field of soil analysis there exists a summary of experiences with this method (VDLUFA, 1997a). These experiences were completed in the last years by additional work and must be continued.

The existing validation, which is done with biowaste and treated biowaste, is not acceptable relating to P, the reproducibility is not satisfactory.

The validation in the next step of the project has to cover all the materials under investigation.

Table 3 Fulfillment of Criteria of the existing standards

Standard Nb.	S=soil SL=sludge W=waste BO= biowaste and treated biowaste, soil improvers	fulfillment of criteria
ISO 11263	S	No
EN 13651	BO	Yes
EN 13652	BO	No
ISO/DIS 21268-2	S	No
ISO/DIS 21268-3	S	No
PrCEN 14429	W	No
EN 12457-1-4	W	No

5. CRITICAL POINT AND RECOMMENDATIONS

The most important point of the proposal of the EN 13651 as a draft standard for the extraction of “soluble P” is that the extraction solution, which has an original pH of 2,65, has a weak buffer capacity. Therefore small changes in the composition of materials under investigation may result in high differences of the extracted amount of P in the extraction solution, especially if the material has limy components and a high pH. The question is if the standard ISO 14870 is the better method which operates under buffered conditions at pH 7,3. The extractability of P decreases at this pH and there is the demand to do a lot of analytical research. Most experiences concerning P exist with the EN 13651 and therefore in the next step the main attention is directed to the ruggedness and the validation of this method.

Next some questions will address the following points that arose in the desk study.

- ratio of sample to extraction solution (solid/liquid)
- shaking frequency and shaking time
- how to analyse samples with low content of dry matter
- the problem of extraction of materials with higher pH, limed or limy materials

6. DRAFT STANDARD

See Annex of this desk study.

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