



SLOVENSKI STANDARD

SIST EN 14672:2005

01-december-2005

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Characterization of sludges - Determination of total phosphorus

Charakterisierung von Schlämmen - Bestimmung von Gesamtphosphor

iTeh STANDARD PREVIEW

Caractérisation des boues - (dosage du phosphore total)

Ta slovenski standard je istoveten z: **EN 14672:2005**

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ICS:

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 14672

August 2005

ICS 13.030.20

English version

Characterization of sludges - Determination of total phosphorus

Caractérisation des boues - Dosage du phosphore total

Charakterisierung von Schlämmen - Bestimmung von
Gesamtposphor

This European Standard was approved by CEN on 27 June 2005.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Contents	Page
Foreword	3
1 Scope	4
2 Normative references	4
3 Terms and definitions	4
4 Principle	5
5 Limitations and interferences	5
6 Hazards	5
7 Apparatus	5
8 Reagents	6
9 Sample pre-treatment	6
10 Procedure	6
11 Expression of results	7
12 Test report	8
Annex A (informative) Performance data of interlaboratory comparison	9

SIST EN 14672:2005

<https://standards.iteh.ai/catalog/standards/sist/5830ec94-2e0f-4d15-a982-23fe597c96a9/sist-en-14672-2005>

Foreword

This European Standard (EN 14672:2005) has been prepared by Technical Committee CEN/TC 308 "Characterization of sludges", the secretariat of which is held by AFNOR.

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 February 2006, and conflicting national standards shall be withdrawn at the latest by February 2006.

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

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EN 14672:2005 (E)**1 Scope**

This European Standard specifies a procedure for complete digestion of raw/digested sludge, suitable for the subsequent determination of phosphorus.

NOTE Phosphorus in environmental samples can be present in many forms. This method of pre-treatment enables the analyst to determine all forms - i.e. total phosphorus, by quantitative oxidation yielding an aqueous solution of orthophosphate which should be analysed for phosphate according to method EN ISO 6878, EN ISO 10304-2 or an equivalent validated method.

The range of the method is up to 50 g/kg P assuming appropriate dilution of the digest in the final measurement step.

2 Normative references

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

EN 12880:2000, *Characterization of sludges — Determination of dry residue and water content*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

EN ISO 6878, *Water quality — Determination of phosphorus — Ammonium molybdat spectrometric method (ISO 6878:2004)*

EN ISO 10304-2, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 2: Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste water (ISO 10304-2:1995)*

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3 Terms and definitions

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

3.1**total phosphorus**

phosphorus converted to orthophosphate by oxidation.

NOTE This includes condensed phosphates (e.g. pyro and polyphosphates), organophosphorus (e.g. phosphate esters and aminophosphonic acids) and free phosphates

3.2**dry residue**

dry mass portion of the sludge obtained after the specified drying process. It is expressed as percent

[EN 12880:2000, 3.1]

3.3**dry mass (dry matter)**

mass of solid obtained after the specified drying process. It is expressed as grams or kilograms

[EN 12880:2000, 3.3]

4 Principle

A boiling mixture of sulfuric and nitric acids oxidises organic matter, releasing any phosphorus present and converting it to orthophosphate. As the mixture boils, nitric acid is lost due to the oxidation process and due to distillation. As the proportion of nitric acid decreases, the boiling temperature rises and any organic matter still unoxidised reacts with the sulfuric acid, causing the mixture to char. Small additions of nitric acid are used to complete the oxidation, at which point, no further charring occurs resulting in white fumes and a clear solution. This solution, when diluted and neutralised with sodium hydroxide, is used for phosphate analysis by method EN ISO 10304-2, EN ISO 6878 or an equivalent validated method. It is possible to analyse for phosphorus using ICP-OES, but the performance of this method shall be shown to be equivalent to the above standard method.

NOTE 1 Many laboratories determine phosphorus together with metals and sulfur using ICP-OES after a suitable nitric acid based digestion, but it is felt that this nitric/sulfuric acid digestion method has been successfully used for many years for total phosphorus determinations and should still be available as an International Standard.

NOTE 2 Determination of P according to EN ISO 10304-2 by ion chromatography can be difficult when sulfate is present at relatively high concentrations in the sample.

5 Limitations and interferences

If fluoride or fluorapatite is known to be present, a thick-walled Kjeldahl flask shall be used.

6 Hazards

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6.1 General

Waste and sludge samples may contain hazardous and inflammable substances. They shall contain pathogens and be liable to biological action. Consequently it is recommended that these samples should be handled with special care. The gases which may be produced by microbiological activity are potentially inflammable and will pressurise sealed bottles. Exploding bottles are likely to result in infectious shrapnel and/or pathogenic aerosols. Glass bottles shall be avoided wherever possible. National regulations should be followed with respect to microbiological hazards associated with this method.

NOTE All equipment used in the vicinity should be flame proof to avoid any source of ignition.

6.2 Storage

It is not advisable to store samples in the open laboratory. If samples are to be stored, store them between 0 °C and 4 °C. Sample containers that allow for the escape of any generated gas should be used.

6.3 Strong acids and alkalis

The sulfuric and nitric acids and the sodium hydroxide, as solid or in solution, are extremely corrosive and shall be handled with caution. Resistant gloves and eye protection shall be worn. Heating of acids shall be carried out under local exhaust ventilation.

7 Apparatus

Ordinary laboratory apparatus and the following:

7.1 Kjeldahl flask, for example 200 ml nominal capacity or tube, for example 100 ml.

7.2 Heating device, Bunsen burner or heating mantle or suitable heating block.

EN 14672:2005 (E)**8 Reagents**

8.1 Sulfuric acid, $\rho = 1,84$ g/ml (18 mol/l).

8.2 Nitric acid, $\rho = 1,42$ g/ml (15 mol/l).

8.3 Sodium hydroxide $c(\text{NaOH}) = (5 \text{ mol/l})$.

Dissolve (200 ± 10) g of sodium hydroxide in approximately 800 ml of water, cool and dilute to $(1\ 000 \pm 10)$ ml with water. Store in a polyethylene bottle.

8.4 Phenolphthalein indicator solution.

8.5 Water, distilled or deionised, complying with grade 3 defined in EN ISO 3696.

9 Sample pre-treatment**9.1 Sample preservation**

Sludge samples can change composition through biological and/or chemical activity. They shall be kept in cool boxes at less than 4°C in transit to the laboratory. Total phosphorus is a conservative parameter. Analysis shall be within 4 weeks.

9.2 Preparation of sample

The purpose of pre-treatment is to obtain a representative and homogeneous sample.

NOTE 1 Sludge may be analysed dried and ground or as a well-homogenised wet sample.

NOTE 2 Dry residue obtained according to EN 12880 may be ground by suitable means (e.g. pestle and mortar) or wet sample which has been macerated or homogenised mechanically may be used directly.

10 Procedure

Weigh sufficient sample (up to $2 \text{ g} \pm 2 \text{ mg}$ dry mass), containing not more than 50 mg of phosphorus into the Kjeldahl flask or tube.

Add $(2,0 \pm 0,1)$ ml sulfuric acid (8.1) and approximately 0,5 ml nitric acid (8.2). Swirl to mix.

Heat the Kjeldahl flask or tube in a fume cupboard.

Continue heating until brown fumes cease to be produced.

If the mixture chars, carefully add approximately 0,5 ml of nitric acid (8.2), dropwise down the neck of the Kjeldahl flask and repeat 3 or 4 times the addition of 0,5 ml of nitric acid until the mixture does not char when white fumes are produced.

Allow the mixture to cool, add 10 ml of water (8.5) and heat to fuming and repeat one time this operation and allow to cool.

Cautiously wash down the sides of the Kjeldahl flask or tube with (25 ± 1) ml of water and allow to cool.

Neutralise the solution with the sodium hydroxide (8.3), in presence of phenolphthalein (8.4).

Quantitatively transfer the solution to a 50 ml volumetric flask, with water, make up to volume and mix well.

Analyse the solution for phosphate phosphorus using method EN ISO 6878, EN ISO 10304-2 or equivalent validated method within 4 weeks.

A reagent blank without the addition of any sludge shall be taken through the whole procedure.

11 Expression of results

11.1 Wet sludge sample

The total phosphorus content (P_{tot}) of the original sample is given in equation (1):

$$P_{\text{tot}} = \frac{(C - C_b) \times V}{m_w \times 10 \times w_{\text{dr}}} \quad (1)$$

where

P_{tot} is the total phosphorus content in grams per kilogram dry matter;

C is the phosphorus concentration of the digestion solution in milligrams per litre;

C_b is the phosphorus concentration in the reagent blank, in milligrams per litre;

V is the volume of the digestion solution in the volumetric flask in millilitres;

m_w is the wet mass of sample taken for digest in grams;

w_{dr} is the dry residue of the sludge determined by method EN 12880 in percent.

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11.2 Dry sludge sample

The total phosphorus content (P_{tot}) of the original sample is given in equation (2):

$$P_{\text{tot}} = \frac{(C - C_b) \times V}{m_D \times 1000} \quad (2)$$

where

P_{tot} is the total phosphorus content in grams per kilogram;

C is the phosphorus concentration of the digest in milligrams per litre;

C_b is the phosphorus concentration in the reagent blank, in milligrams per litre;

V is the volume of solution in volumetric flask in millilitres;

m_D is the dry mass of sample taken for acid digestion in grams.

11.3 Precision

See Annex A.