SLOVENSKI STANDARD
SIST EN 15959:2012

01-marec-2012

Gnojila - Določevanje ekstrahiranega fosforja

Fertilizers - Determination of extracted phosphorus

Düngemittel - Bestimmung von Phosphor in den Extrakten

Engrais - Dosage du phosphore extrait

Ta slovenski standard je istoveten z: **EN 15959:2011**

ICS:

65.080	Gnojila	Fertilizers

SIST EN 15959:2012 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15959

November 2011

ICS 65.080

Supersedes CEN/TS 15959:2009

English Version

Fertilizers - Determination of extracted phosphorus

Engrais - Dosage du phosphore extrait

Düngemittel - Bestimmung von Phosphor in den Extrakten

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Ref. No. EN 15959:2011: E

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Foreword

This document (EN 15959:2011) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by May 2012, and conflicting national standards shall be withdrawn at the latest by May 2012.

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 15959:2009.

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

The following has been added to the former edition of the European Standard:

a) the CEN Technical Specification has been adopted as a European Standard;

b) editorial revision.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination of phosphorus in fertilizer extracts.

The method is applicable to all extracts of fertilizers for the determination of the different forms of phosphorus as phosphorus soluble in mineral acids, water soluble phosphorus, phosphorus soluble in solutions of ammonium citrate, phosphorus soluble in 2 % citric acid and phosphorus soluble in 2 % formic acid.

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 1482-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

EN 12944-1:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 2: Terms relating to fertilizers*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1:1999 and EN 12944-2:1999 apply.

4 Principle

After possible hydrolysis, phosphorus is precipitated in an acid media in the form of quinoline phosphomolybdate.

After filtering and washing, the precipitate is dried at 250 °C and weighed.

In the above mentioned conditions no interfering action is exerted by the compounds likely to be found in the solution (mineral and organic acids, ammonium ions, soluble silicates, etc.) if a reagent based on sodium molybdate or ammonium molybdate is used in the precipitation.

5 Sampling and sample preparation

Sampling is not part of the method specified in this document. A recommended sampling method is given in EN 1482-1.

Sample preparation shall be carried out in accordance with EN 1482-2. Grinding of the laboratory sample is recommended for homogeneity reasons.

6 Reagents

6.1 Water, distilled or demineralized.

6.2 Concentrated nitric acid, ρ = 1,40 g/ml.

6.3 Preparation of reagents

6.3.1 Preparation of the reagent based on sodium molybdate

Solution A: Dissolve 70 g of sodium molybdate dihydrate in 100 ml of distilled water.

Solution B: Dissolve 60 g of citric acid monohydrate in 100 ml of distilled water and add 85 ml concentrated nitric acid (6.2).

Solution C: Stir solution A into solution B to obtain solution C.

Solution D: To 50 ml of distilled water, add 35 ml of concentrated nitric acid (6.2), then 5 ml of freshly distilled quinoline. Add this solution to solution C, mix thoroughly and leave standing overnight in the dark. After this make up to 500 ml with distilled water, mix again, and filter through a sintered glass funnel (7.7).

6.3.2 Preparation of the reagent based on ammonium molybdate

Solution A: In 300 ml of distilled water, dissolve 100 g of ammonium molybdate while heating gently and stirring from time to time.

Solution B: Dissolve 120 g of citric acid monohydrate in 200 ml of distilled water, add 170 ml of concentrated nitric acid (6.2).

Solution C: Add 10 ml of freshly distilled quinoline to 70 ml of concentrated nitric acid (6.2).

Solution D: Slowly pour, stirring well, solution A into solution B. After thoroughly mixing add solution C to this mixture and make up to 1 l. Leave standing for two days in a dark place and filter through a sintered glass funnel (7.7).

The reagents 6.3.1 and 6.3.2 may be used in the same way; both shall be kept in the dark in stoppered polyethylene bottles.

7 Apparatus

7.1 Standard laboratory equipment.

7.2 500 ml Erlenmeyer flask, with a wide neck.

7.3 Graduated pipettes, of 10 ml, 25 ml and 50 ml.

7.4 Filter crucible, with porosity of 5 µm to 20 µm.

7.5 Buchner flask.

7.6 Drying oven, regulated at (250 ± 10) °C.

7.7 Sintered glass funnel, with porosity of 5 µm to 20 µm.

8 Procedure

8.1 Treatment of the solution

With a pipette (7.3), take an aliquot part of fertilizer extract (see Table 1) containing about 0,01 g of P_2O_5 and place it in the Erlenmeyer flask (7.2). Add 15 ml of concentrated nitric acid (6.2) and dilute with water to about 100 ml. When the solution to be precipitated contains more than 15 ml of citrate solution (neutral citrate, Petermann or Joulie alkaline citrate), an amount of 21 ml of concentrated nitric acid (6.2) shall be added.

Table 1 — Determination of the aliquot parts of the phosphate solutions

% P_2O_5 in the fertilizer	% P in the fertilizer	Sample for analysis g	Dilution to ml	Sample ml	Dilution to ml	Sample to be precipitated ml	Quinoline phospho-molybdate conversion factor (F), in % P_2O_5	Quinoline phospho-molybdate conversion factor (F'), in % P
5 to 10	2,2 to 4,4	1	500	-	-	50	32,074	13,984
		5	500	-	-	10	32,074	13,984
10 to 25	4,4 to 11,0	1	500	-	-	25	64,148	27,968
		5	500	50	500	50	64,148	27,968
> 25	> 11	1	500	50	500	10	160,370	69,921
		5	500	50	500	25	128,296	55,937

8.2 Hydrolysis

If the presence of metaphosphates, pyrophosphates or polyphosphates is suspected in the solution, carry out the hydrolysis as follows.

Bring the content of the Erlenmeyer flask (7.2) to the boil slowly and keep at this temperature until hydrolysis is completed. This usually takes 1 h. Care shall be taken to avoid losses by splashing and excessive evaporation which could reduce the initial volume by more than half, by fitting a reflux condenser. After hydrolysis make up to the initial volume with water (6.1).

8.3 Weighing the crucible

Dry the filter crucible (7.4) for at least 15 min in the drying oven (7.6) set at (250 ± 10) °C. Cool in a desiccator and weigh.

8.4 Precipitation

Heat the acid solution contained in the Erlenmeyer flask (7.2) until it begins to boil. Then start the precipitation of the quinoline phosphomolybdate by adding 40 ml of the precipitating reagent (6.3.1 or 6.3.2) drop by drop, stirring continuously. To precipitate phosphate solutions containing more than 15 ml of citrate solution (neutral citrate, Petermann or Joulie) which have been acidified with 21 ml of concentrated nitric acid (see 8.1), an amount of 80 ml of the precipitating reagent shall be used. Place the Erlenmeyer flask in a steam bath, leave it there for 15 min, shaking it from time to time. The solution may be filtered immediately or after it has cooled down.

8.5 Filtering and washing

Filter the solution under vacuum by decantation. Wash the precipitate in the Erlenmeyer flask (7.2) with 30 ml of water (6.1). Decant and filter the solution. Repeat this process five times. Quantitatively transfer the rest of the precipitate into the crucible washing it with water. Wash four times with 20 ml of water, allowing the liquid to drain from the crucible before each addition. Dry the precipitate thoroughly.

8.6 Drying and weighing

Wipe the outside of the crucible with a filter paper. Place this crucible in a drying oven (7.6) and keep it there until its mass remains constant, at a temperature of 250 °C (usually 15 min); leave it to cool in the desiccator at ambient temperature and weigh rapidly.

8.7 Blank test

For each series of determinations, carry out a blank test using only the reagents and solvents in the proportions used in the extraction (citrate solution, etc.) and allow for them in the calculation of the final result.

8.8 Verification

Carry out the determination using an aliquot part of a potassium dihydrogen phosphate solution containing 0,01 g of P_2O_5.

9 Calculation and expression of the result

If the samples for analysis and dilutions are used according to Table 1, calculate the mass fraction, w_P, in percent according to Equation (1):

$$w_P = (m_1 - m_2) \times F' \quad (1)$$

If the samples for analysis and dilutions are used according to Table 1, calculate the mass fraction, $w_{P_2O_5}$, in percent according to Equation (2):

$$w_{P_2O_5} = (m_1 - m_2) \times F \quad (2)$$

where

m_1 is the mass, in grams, of the quinoline phosphomolybdate;

m_2 is the mass, in grams, of the quinoline phosphomolybdate obtained in the blank test;

F and F' are factors given in the last two columns of Table 1.

If the samples for analysis and dilutions differ from those of Table 1, calculate the mass fraction, w_P, in percent according to Equation (3):

$$w_P = \frac{(m_1 - m_2) \times f' \times D \times 100}{M} \quad (3)$$

If the samples for analysis and dilutions differ from those of Table 1, calculate the mass fraction, $w_{P_2O_5}$, in percent according to Equation (4):

$$w_{P_2O_5} = \frac{(m_1 - m_2) \times f \times D \times 100}{M} \quad (4)$$