

LABORATORY MANAGEMENT SYSTEM
OF THE DEPARTMENT OF ENVIRONMENT PROTECTION
MARITIME INSTITUTE, GDYNIA MARITIME UNIVERSITY



Edition No. 3
Date 05.10.2019
Page/Pages: 1/7

RESEARCH PROCEDURE PB-44A

**DETERMINATION OF AVAILABLE PHOSPHORUS
IN SOIL WATER EXTRACT**

Supervised document

Developed by:

Agnieszka Cichowska

Opined by:

Katarzyna Szczepańska

Approved by:

Quality Manager

Quality Manager

Grażyna Pazikowska-Sapota

Grażyna Dembska

LABORATORY MANAGEMENT SYSTEM
OF THE DEPARTMENT OF ENVIRONMENT PROTECTION
MARITIME INSTITUTE, GDYNIA MARITIME UNIVERSITY



Research Procedure PB-44A

Determination of available
phosphorus
in soil water extract

Edition No. 3
Date 05.10.2019
Update 0
Page/Pages: 2 /7

TABLE OF CONTENTS

1. PURPOSE
2. SUBJECT - SCOPE
3. PERSONNEL QUALIFICATIONS
4. DESIGNATION
5. REAGENTS AND SUPPORT MATERIALS
6. EQUIPMENT
7. TEST CONDITIONS
8. DESCRIPTION OF THE PROCEDURE
 - 8.1. Soil sampling
 - 8.2. Preparation of the test sample
 - 8.3. Stages of proceedings
 - 8.4. Expression of results
 - 8.5. Quality assurance
 - 8.6. Control and finding of irregularities in tests
9. LIST OF REFERENCED DOCUMENTS
 - 9.1. Normative documents
 - 9.2. Laboratory Management System documents
10. ADDITIONAL INFORMATION

LABORATORY MANAGEMENT SYSTEM
OF THE DEPARTMENT OF ENVIRONMENT PROTECTION
MARITIME INSTITUTE, GDYNIA MARITIME UNIVERSITY



Research Procedure PB-44A

Determination of available
phosphorus
in soil water extract

Edition No. 3
Date 05.10.2019
Update 0
Page/Pages: 3 /7

1. PURPOSE

The purpose of the procedure is to determine the method for analysis the phosphorus content in an water extract prepared from agricultural soil, by atomic emission spectrometry with excitation in inductively coupled plasma (ICP-OES).

2. SUBJECT - SCOPE

The subject of the study are soils.

Lower limit of quantification: 1,0 mg·kg⁻¹.

3. PERSONNEL QUALIFICATIONS

The employee conducting the study should have: secondary chemical education and analytical experience.

4. DESIGNATION

Phosphorus (P_{H2O}).

5. REAGENTS AND SUPPORT MATERIALS

- Deionized water
- Phosphorus standard solution dedicated for ICP-OES

6. EQUIPMENT

- Inductively excited plasma optical emission spectrometer (ICP-OES)
- Shaker
- Centrifuge
- Technical weight
- 50 cm³ polypropylene containers, suitable for centrifugation
- Automatic pipettes with a capacity of 1 and 10 cm³
- Laboratory dryer

7. TEST CONDITIONS

The tests are carried out at ambient temperature.

8. DESCRIPTION OF THE PROCEDURE

8.1 Soil sampling

- take a general sample from a 0-30 cm layer of soil

LABORATORY MANAGEMENT SYSTEM
OF THE DEPARTMENT OF ENVIRONMENT PROTECTION
MARITIME INSTITUTE, GDYNIA MARITIME UNIVERSITY



Research Procedure PB-44A

Determination of available
phosphorus
in soil water extract

Edition No. 3
Date 05.10.2019
Update 0
Page/Pages: 4 /7

- to take a general sample, up to 20 incremental (single) samples should be taken evenly from the field surface
- the general (average) sample should represent the area of agricultural use with similar natural conditions (i.e. soil type, terrain type) and agrotechnical conditions (fore crop, cultivation, fertilization)
- the usable area per general sample, with an aligned soil surface and similar terrain, may not exceed 4 ha.
- the general sample should be prepared separately for each crop.
 - general samples should be marked on a precisely made terrain sketch and numbered respectively to the field surface they represent. samples taken from grassland must be marked with X in addition to the number.

8.2 Preparation of the test sample

- the sample should be stored at 2–5°C until analysis
- the soil sample after quartering should be dried (air drying according to PPN-ISO 11464:1999).

8.3 Stages of proceedings

Determination of humidity (according to Research Procedure PB-11 edition No. 4)

- a) Dry the opened weighing container in the laboratory dryer for 1 h at 105°C
- b) Close the dry container and place in a desiccator to cool to room temperature (approx. 0.5 h)
- c) Weigh the empty container
- d) Weigh in container approximately 10 g of dry soil
- e) Dry opened container with soil for 2 hours in a laboratory dryer at 105°C
- f) Close the container with soil and place in a desiccator to cool to room temperature (approx. 0.5 h)
- g) Weigh the container with soil
- h) Calculate the humidity from the following formula:

$$W (\%) = \frac{(mm - ms) * 100\%}{mm}$$

W - humidity (%)

mm – soil wet weight

ms - soil dry weight

LABORATORY MANAGEMENT SYSTEM
OF THE DEPARTMENT OF ENVIRONMENT PROTECTION
MARITIME INSTITUTE, GDYNIA MARITIME UNIVERSITY



Research Procedure PB-44A

Determination of available
phosphorus
in soil water extract

Edition No. 3
Date 05.10.2019
Update 0
Page/Pages: 5 /7

- i) the operations described in points e) to h) should be repeated until constant mass of the sample is reached (i.e. 0.5% of the last result).

Extraction

- Weigh 0.5 g of dry and ground sample (after humidity determination) into polypropylene container
- Pour a sample of 25 cm³ distilled water
- Shake the sample for 30 min
- Centrifuge sample for 15 min at 4 rpm
- Transfer the supernatant to a new container.

Analysis

- Determine in the supernatant the phosphorus content by atomic emission spectrometry with excitation in inductively coupled plasma (ICP-OES).

8.4 Expression of results

P_{H_2O} concentration is given in g P·kg⁻¹ dry weight of sample.

8.5 Quality assurance

In order to ensure the quality and check the measuring system (reagents, measuring device, sample preparation) and the sample handling mode, a phosphorus standard solution 1000 µg·cm⁻³ is used. The standard solution stored in a polypropylene container at room temperature is stable for about 6 months. It is possible to dilute a given standard with water depending on the expected results of the samples.

Parallel samples should also be analysed (every 10 samples per measurement series) and control standard (once in a measuring series).

The method of standard addition can be used to determine the characteristic interactions for a given sample (matrix effect).

LABORATORY MANAGEMENT SYSTEM
OF THE DEPARTMENT OF ENVIRONMENT PROTECTION
MARITIME INSTITUTE, GDYNIA MARITIME UNIVERSITY



Research Procedure PB-44A

Determination of available
phosphorus
in soil water extract

Edition No. 3
Date 05.10.2019
Update 1
Page/Pages: 6 /7

8.6 Control and finding of irregularities in tests

Test control or verification must be carried out in accordance with the program for monitoring the validity of measurements results in accordance with Management System Book. Irregularities in tests found by the Staff or the Head of the Laboratory cause the repeated analysis by an authorized person.

9. LIST OF REFERENCED DOCUMENTS

- Pietrzak S., Majewska Z., Wesołowski P., 2016. Przydatność wskaźnika wysycenia gleby fosforem do oceny ryzyka wynoszenia tego składnika w spływie do wód powierzchniowych- studium przypadku, Woda-Środowisko-Obszary Wiejskie, T. 16 Z. 2 (54), 89-98, ISSN 1642-8145

9.1 Normative documents

- PN-ISO 11464:1999 - Polish version. Soil quality - Initial sample preparation for physico-chemical tests
- PN-R-04031:1997 – Polish version. Chemical and agricultural analysis of soil. Sampling.

9.2 Laboratory Management System documents

Management System Book.

10. ADDITIONAL INFORMATION

Not included.

Verification:

S. Pietrzak, G. Pazikowska-Sapota, G. Dembska, L. Dzierzbicka-Głowacka, M. Urbaniak, Z. Majewska, D. Juszkowska, A. Cichowska. Risk of phosphorus removal in surface runoff from agricultural land of the Baltic commune of Puck - presentation proposal The 9th International Phosphorus Workshop nt. "Putting phosphorus first? How to address current and future challenges", 8-12 lipca 2019 r., Zurich

Ostrowska D., Cichowska A., Szczepańska K., Dembska G., Pazikowska-Sapota G., Galer-Tatarowicz K., Chmielewska J., Bojke A., Dzierzbicka-Głowacka L. Determination of absorbable forms of potassium and magnesium in agricultural land of the Baltic commune of Puck by the ICP-OES method, II Scientific Conference of Polish Sea Researchers, Gdynia, 24-25.09.2019, Abstracts book – P_056, p. 186 - Poster

LABORATORY MANAGEMENT SYSTEM
OF THE DEPARTMENT OF ENVIRONMENT PROTECTION
MARITIME INSTITUTE, GDYNIA MARITIME UNIVERSITY



Research Procedure PB-44A

Determination of available
phosphorus
in soil water extract

Edition No. 3
Date 05.10.2019
Update 0
Page/Pages: 7 /7

Pazikowska-Sapota G., Pietrzak S., Dembska G., Ostrowska D., Cichowska A., Juszkowska D., Majewska Z., Urbaniak M., Galer-Tatarowicz K., Dzierzbicka-Głowacka L. Risk assessment of phosphorus removal in surface runoff from the lands of the Baltic PUCK commune, II Scientific Conference of Polish Sea Researchers, Gdynia, 24-25.09.2019, Abstracts book - R_053, p. 92 – Presentation

S. Pietrzak, G. Pazikowska-Sapota, G. Dembska, L. Dzierzbicka-Głowacka, D. Juszkowska, Z. Majewska, M. Urbaniak, D. Ostrowska, A. Cichowska, K. Galer-Tatarowicz. 2020. Risk of phosphorus losses in surface runoff from agricultural land in the Baltic Commune of Puck in the light of assessment performed on the basis of DPS indicator. PeerJ 8:e8396 <https://doi.org/10.7717/peerj.8396>