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## Mineral nitrogen in soils of Lithuania's agricultural land: comparison of oven-dried and field-moist samples

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### Abstract

Mineral nitrogen ( $N_{\min} = N-NH_4^+ + N-NO_3^-$ ) in oven-dried and field-moist samples was investigated in 2011–2014 in five areas of Lithuania with soils of different texture (sand, sandy loam, loam and clay) and different mineral nitrogen ( $N_{\min}$ ) content. The samples were taken in spring (from late March to early April) and autumn (late October – early November), from the 0–30, 30–60 and 60–90 cm as well as from 0–60 cm soil layers. The aim of the research was to determine the influence of different methods of soil sample preparation on the concentrations of ammonium ( $N-NH_4^+$ ) and nitrate ( $N-NO_3^-$ ) nitrogen in oven-dried and field-moist samples and to assess the suitability of the obtained results for the development of agricultural plant fertilisation plans.

According to the research results, drying of soil samples at a temperature of 35°C for 12 hours resulted in significantly higher levels of  $N-NH_4^+$  compared to those of field-moist samples. The  $N-NO_3^-$  concentrations in the field-moist samples, when stored at a 4°C temperature for 3 days, increased marginally compared to the oven-dried soil samples. Weak or moderate, however, statistically significant correlations were observed between  $N-NH_4^+$  concentrations in the oven-dried and field-moist samples. In order to perform faster chemical analyses of soil  $N_{\min}$  for making agricultural plant fertilisation plans, it is necessary to carry out analyses on oven-dried soil samples, and using the linear correlation equations, to convert the results of  $N-NH_4^+$  analyses into field-moisture data.

Keywords: ammonium and nitrate nitrogen, preparation of samples for analysis.

### Introduction

Nitrogen (N) is vital for all living organisms and it is of utmost importance for the increase of agricultural productivity, which facilitates solving the problems of supplying human population with food globally to a large extent (Prud'homme, 2005; Wiesler, Armbruster, 2009).

Although nitrogen is one of the key factors shaping fertility of soil and productivity of plants, its abundance can cause damage to the environment (Füleky, 2009; Rutkowska, Fotyma, 2009). By virtue of an intensive use of nitrogen fertilisers, where fertiliser rates exceed plant needs, and in the case of other nutrient shortage in soil the nitrates unused by plants enter surface and ground waters causing pollution (Bouwman et al., 2005; Adomaitis et al., 2008; Eriksen et al., 2008). All of this may have a negative impact both on the health status of people and wildlife (WHO, 1985). Therefore, in 1991 the European Commission published the Nitrates Directive (91/676/EEC) concerning the protection of waters against pollution caused by nitrates from agricultural sources, which prescribes the limit of groundwater pollution contamination by nitrates – 50 mg l<sup>-1</sup> (Council Directive, 1991).

After harvesting,  $N-NO_3^-$  that was not absorbed by plants and remaining in soil is identified as the main source of nitrate leaching in winter (Bogaert et al., 2000).

Therefore, it is imperative, that in autumn  $N-NO_3^-$  quantities in soil are as low as possible and its leaching to ground water is not increased (Rutkowska, Fotyma, 2009).

In order to reduce nitrate leaching, in Lithuania, Germany, Poland and other European countries nitrogen fertiliser rates for agricultural crops are determined taking account of mineral nitrogen ( $N_{\min}$ ) levels present in soil in spring. Soil samples are taken in autumn in order to determine the concentrations of these compounds remaining in soil after harvesting (Staugaitis et al., 2007; Arbačiauskas et al., 2014).

Mineral nitrogen concentration in a specific soil layer in real place and time reflects the balance of nitrogen released during organic matter mineralisation as well as that incorporated with mineral and organic fertilisers and fixed by microorganisms on the one hand, and nitrogen assimilated by plants, leached, denitrified and immobilised on the other hand (Wong, Nortcliff, 1995).

Therefore, it is very important to take soil samples properly and prepare them for chemical analyses. If soil samples are taken from soils untypical for the area and they are inadequately prepared, chemical analyses do not indicate accurate  $N_{\min}$  concentrations in the analysed field soil (Anderson, Ingram, 1993). To prepare an average soil sample is complicated as in outdoor conditions such

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sample is composed of 10–15 borings and for chemical analyses only 25 g of soil is required. Thus, in preparing an average soil sample it is easier, and chemical analyses can be conducted faster to mix oven dried soil compared to field-moist soil (Vandendriessche et al., 2011; Deressa, 2014). This is particularly important since intensive biochemical transformation of nitrogen compounds takes place in soil: ammonification, nitrification, fixation in soil sorption complex, denitrification and evaporation depending on soil moisture, temperature and other factors. It is also noted that sample drying affects the results of ammonium and nitrate analyses (Gray, McLaren, 2003; Nina, Sigunga, 2012).

Soil sample storage prior to chemical analyses may still allow continuation of ammonification and denitrification processes in soil depending on storage conditions (Gray, McLaren, 2003; Nina, Sigunga, 2012). Nitrogen concentrations in soil and fertilisation recommendations for farmers, therefore, have to be carried out as soon as possible (Mengel, Kirkby, 2001; Staugaitis et al., 2007). Studies relating to the influence of soil sample preparation for chemical analyses on mineral nitrogen ( $N_{\min} = N-NH_4^+ + N-NO_3^-$ ) concentrations in soil are rarely found in scientific literature, and they have never been carried out under the conditions of our country.

**Table 1.** Soil agrochemical characteristics of study sites

No.	Coordinates of study site	$pH_{KCl} \pm SD$	Soil organic carbon*	Total nitrogen**	Mineral nitrogen
			% $\pm$ SD		mg kg <sup>-1</sup> $\pm$ SD
1.	<i>x 409428, y 6158486</i>	6.4 $\pm$ 0.27	2.57 $\pm$ 0.22	0.200 $\pm$ 0.078	7.41 $\pm$ 3.56
2.	<i>x 435370, y 6062869</i>	6.9 $\pm$ 0.32	1.31 $\pm$ 0.27	0.128 $\pm$ 0.046	5.73 $\pm$ 1.31
3.	<i>x 574244, y 6158473</i>	5.1 $\pm$ 0.47	0.85 $\pm$ 0.24	0.084 $\pm$ 0.007	5.94 $\pm$ 3.18
4.	<i>x 484218, y 6159972</i>	6.6 $\pm$ 0.30	1.56 $\pm$ 0.15	0.160 $\pm$ 0.018	10.8 $\pm$ 4.02
5.	<i>x 573594, y 6158511</i>	5.3 $\pm$ 0.42	1.25 $\pm$ 0.12	0.120 $\pm$ 0.013	9.92 $\pm$ 3.42

SD – standard deviation; \* – ISO 10694:1995. Soil quality - Determination of organic and total carbon after dry combustion, \*\* – ISO 11261:1995. Soil quality - Determination of total nitrogen by modified Kjeldahl method

Soil samples were taken in spring (end of March – beginning of June) and autumn (end of October – beginning of November) at depths of 0–30, 30–60 and 60–90 cm as well as from the 0–60 cm soil layer from 10  $\times$  10 m size sites (in the case of agricultural crop fertilisation experiments – from 6  $\times$  12 m size sites). Each soil sample was composed of 10 borings, homogenised well, packed in a plastic bag and stored at 4°C for three days. According to ISO 14256-2:2005 (Soil quality - Determination of nitrate, nitrite and ammonium in field-moist soils by extraction with potassium chloride solution. Part 2. Automated method with segment flow analysis), this is the maximum permitted storage time for soil samples.

Upon arrival to the laboratory, the field-moist soil samples were prepared by moist soil homogenizer “Vaichenstefen” No. 69/26.5.4899 (Germany). After that the sample was placed on the table and shaped into a square and then diagonally divided into four sections. The sample portion made of two opposite square sections was used for ammonium ( $N-NH_4^+$ ) and nitrate ( $N-NO_3^-$ ) nitrogen determination in the field moisture condition. This sample portion was weighed by a standard operating procedure SVP D-10:2016. Meanwhile, the other part of the sample was poured back into the same plastic bags and used for the analysis of the mentioned elements having dried the soil beforehand. Prior to the analyses to be carried out on the oven-dried samples they had been dried at 35°C for at least 12 hours. The soil dried in the oven was ground, sieved through a 2 mm sieve and a 20 g sample was taken from it for chemical analysis.

Experience has shown that different  $N_{\min}$  determination methods can yield discrepant results.

Therefore, the aim of this work is to determine the influence of different methods of soil sample preparation on the concentrations of ammonium ( $N-NH_4^+$ ) and nitrate ( $N-NO_3^-$ ) nitrogen in oven-dried and field-moist samples and to assess their suitability for preparing agricultural plant fertilisation plans in different soils of Lithuania's agricultural land.

## Materials and methods

The studies were conducted in 2011–2014 in five locations (Table 1). Three of them (No. 1, 2 and 3) were agricultural land areas located in three parts of Lithuania-Western, Central and Eastern. 5–7 sites were selected in each of the aforementioned areas 5–7 on the soils of different types and different texture (sand, sandy loam, loam and clay). For other two (No. 4 and 5) research sites, the data from the experiments carried out in Central and Eastern Lithuania on agricultural crop fertilisation were used. Soil texture in the subsoil of all investigated sites was heavier compared to the arable soil layer. The agrochemical characteristics of the soil from the study sites are presented in Table 1.

The determination of  $N-NH_4^+$  and  $N-NO_3^-$  concentrations in soil samples was performed in accordance with ISO 14256-2:2005. The field-moist samples were analysed using a soil extractant ratio of 1:5. 40 g soil was weighed into a 500 ml plastic bottle and 200 ml 1 mol l<sup>-1</sup> KCl added. Next, the samples were shaken in a shaker for 1 hour at ambient temperature 20  $\pm$  2°C. Then, the suspension was strained using a paper filter into test tubes, decanting the first drops of the filtrate (about 20 ml). The paper filter contamination by nitrogen compounds was verified. Next, the extraction solution was filtered through a filter paper into test tubes. The determined nitrogen compounds were compared with the extraction solution and with their concentrations in the extraction solution. In parallel with the sample analysis the blank test was carried out in accordance with the same procedure using the same quantities of all reagents as in the sample analysis only without the experimental material (soil).

Soil and extractant ratio used for the analysis of the oven-dried samples was also 1:5. 20 g of soil were weighed into a 250 ml plastic bottle. The measure of 100ml l<sup>-1</sup> KCl was taken and added. Subsequently, the same analysis procedure as described for determining nitrogen concentration in the field-moist samples was applied.

Next,  $N-NH_4^+$  and  $N-NO_3^-$  concentrations were analysed using analyser Fiastar 5000 (FOSS Analytical AB “Kongas”, Sweden). The measurements were carried out immediately after the filtrate preparation by this device under its instructions for use. Special cassettes with appropriate filters were used for  $N-NO_3^-$  and  $N-NH_4^+$  determination.

The mineral nitrogen ( $N_{\min}$ ) data derived from the analyses of the field-moist soil samples were converted to dry moisture according to the following formula:

$$y_1, y_2 = \left[ \frac{x_1 : x_2}{m} \right] \times \left[ \frac{v}{m} \times \left( 1 + \frac{w_w}{100} \right) + \frac{w_w}{100} \right],$$

where  $y_1$  is  $N-NH_4$  concentration ( $mg\ kg^{-1}$ ) conversion into dry soil,  $y_2$  –  $N-NO_3$  concentration ( $mg\ kg^{-1}$ ) conversion into dry soil,  $x_1$  –  $N-NH_4$  concentration ( $mg\ kg^{-1}$ ) in field-moist soil,  $x_2$  –  $N-NO_3$  concentration ( $mg\ kg^{-1}$ ) in field-moist soil,  $m$  – soil sample weight used for the extraction (40 g),  $V$  – extraction solution volume (200 ml),  $w_w$  – soil water content by weight % calculated for dry soil.

**Statistical analysis.** The experimental data on  $N-NH_4^+$  and  $N-NO_3^-$  was statistically processed using software *Microsoft Office Excel 2010*. Mean  $N-NH_4^+$  and  $N-NO_3^-$  concentrations and their ratios as well as standard deviations (SD) were calculated. To assess the significance of a two-sample average  $t$  test was applied. At a selected probability level ( $p \leq 0.01$  and  $p \leq 0.05$ ) a factual  $t_{\text{fact}}$  criterion value was compared with a theoretical  $t_{\text{theor}}$  value and the difference between the two means was recognised as significant, when  $t_{\text{fact}} \geq t_{\text{theor}}$ . To determine the strength and nature of the relationship between the variables, correlation and regression data analysis was performed using software *STATISTICA 7* (Hill, Levicki, 2005).

**Table 2.** Ammonium ( $N-NH_4^+$ ) and nitrate ( $N-NO_3^-$ ) nitrogen concentrations in oven-dried and field-moist samples of different texture soils

Depth cm	$N-NH_4^+$		$N-NO_3^-$	
	oven-dried samples			
	field-moist samples			
	$mg\ kg^{-1}$	ratio	$mg\ kg^{-1}$	ratio
Heavy texture soils: loams, clays (cases 432)				
0–30	2.64**	1	5.64	1
	1.04	0.39	6.82**	1.20
30–60	1.65**	1	3.83	1
	0.87	0.52	4.37*	1.14
60–90	1.52**	1	2.65	1
	0.87	0.57	3.01	1.13
Light texture soils: sands, sandy loams (cases 87)				
0–30	2.14**	1	4.11	1
	0.78	0.36	4.63	1.13
30–60	1.33**	1	2.69	1
	0.70	0.52	3.28	1.22
60–90	1.17**	1	1.86	1
	0.71	0.60	2.11	1.14
All soils (cases 519)				
0–30	2.56**	1	5.39	1
	1.00	0.39	6.45**	1.20
30–60	1.59**	1	3.64	1
	0.84	0.52	4.19**	1.15
60–90	1.46**	1	2.52	1
	0.84	0.58	2.86*	1.13

Note. \*\* – statistically significant differences between  $N-NH_4^+$  or  $N-NO_3^-$  concentrations determined in oven-dried and field-moist samples at the 99% probability ( $p \leq 0.01$ ); \* – differences statistically significant at the 95% probability ( $p \leq 0.05$ ).

moist samples were statistically significantly higher compared to the concentration of this element in the oven dried samples (Table 2). However, these changes were more pronounced in the soils of heavier texture compared to light sands of sandy loams.

In Belgium, the researchers found that the sample preparation method to establish  $N-NO_3^-$  concentrations did not influence the determination itself, when the samples were analysed immediately on arrival from the field, storing them at 4°C. However, it confirmed the

## Results and discussion

Ammonification is organic nitrogen transformation into  $NH_4^+$  involving soil microorganisms (Galloway et al., 2004). With a temperature increase the intensity of ammonification increases as well (Sparling, Ross, 1988).

Having analysed ammonium nitrogen ( $N-NH_4^+$ ) concentrations in all of our studied soils we established significantly higher levels of it in the oven-dried samples compared to the field-moist samples, which is contrary in the case of nitrate ( $N-NO_3^-$ ) (Table 2). The concentrations of this element found in the field-moist samples at 0–30, 30–60 and 60–90 cm depths were 1.56, 0.75 and 0.62  $mg\ kg^{-1}$  higher, respectively compared to the oven-dried samples. The  $N-NH_4^+$  concentration ratio between the oven-dried and field-moist samples at the upper (0–30 cm) soil layer was lower (1:0.39) and in the deeper 30–60 and 60–90 cm layers it was higher (1:0.52 and 1:0.58, respectively). In the case of light texture soils,  $N-NH_4^+$  concentration ratio between the oven-dried and field-moist samples at the 0–30 cm layer was insignificantly lower, while at the 60–90 cm depth it was higher, respectively, compared to heavy texture soils.

Our studies found that even when the samples had been stored at 4°C for three days prior to chemical analyses, the nitrification processes continued, therefore, the nitrate nitrogen ( $N-NO_3^-$ ) concentrations in the field-

tendencies achieved by us, that longer storage time of soil samples, even at 4°C, allows further biological oxidation of ammonium to nitrate (nitrification) (Vandendriessche et al., 2011). There is evidence that biological nitrification processes in soil take place even when soil samples are stored at –10°C (Magesan et al., 2002; Nina, Sigunga, 2012). Meanwhile, soil sample drying stops this processes in soil (Vandendriessche et al., 2011). In moist soil oxygen stimulates the activity of nitrifying bacteria resulting in an increased nitrate ( $NO_3^-$ ) content

in the samples during their storage. This is determined by oxygen access to the soil during sample preparation and storage. As Alvarez and Steinbach (2009) indicate soil tillage results in improved soil aeration, which influences the increase in  $\text{N-NO}_3^-$  content compared to untilled soil. This can also be applied in the case of soil sampling, when more oxygen enters soil.

The concentrations of  $\text{N-NO}_3^-$  distributed in a consistently decreasing sequence at different (0–30, 30–60 and 60–90 cm) soil layers, and in deeper soil layers it was lower compared to the top 0–30 cm layer. The concentrations of  $\text{N-NO}_3^-$  in the field-moist samples at depths of 0–30, 30–60 and 60–90 cm were 1.06, 0.55 and 0.34  $\text{mg kg}^{-1}$  higher, respectively, compared to the concentrations of this element established in the oven-dry samples. The dependence tendencies of this element concentrations on the soil layer depth expressed in relative numbers was the same as that assessed in actual figures. If the ratio between  $\text{N-NO}_3^-$  concentrations in the oven-dried and field-moist samples at the 0–30 cm soil layer was 1.20, deeper 30–60 and 60–90 cm layers demonstrated ratios of 1.15 and 1.13, respectively.

In the case of heavier texture soils, the ratios of the established  $\text{N-NO}_3^-$  concentrations in the field-moist samples and the concentrations of this element in the oven-dry samples as well as the variation tendencies achieved were the same as in overall assessment of all the soils.

The  $\text{N-NO}_3^-$  concentrations in light textured soils were lower compared to those in heavier textured soils.  $\text{N-NO}_3^-$  determined in the field-moist samples was also higher compared to the dried samples; however, the differences achieved between the samples were not significant. These tendencies were observed in all soil layers.

The data presented in Table 3 indicate that the correlation-regression data analysis showed that the dependence of  $\text{N-NH}_4^+$  concentrations in the field-moist samples ( $y_2$ ) on the concentrations of this element in the oven-dried samples ( $x_2$ ) taken from the 0–30 and 30–60 cm soil layers was weak ( $r = 0.300$  and  $0.417$ ), and that from the 60–90 cm was moderately strong ( $r = 0.564$ ). However, in all cases the correlations were statistically significant at a 99% probability ( $p \leq 0.01$ ).

**Table 3.** Relationship between ammonium nitrogen ( $\text{N-NH}_4^+$ ) concentrations in field-moist samples ( $y$ ) and their concentrations in oven-dried samples ( $x$ ) in different soil layers

Soil layer cm (cases 519)	Parameters of equation		Correlation coefficient
	$y = a + bx$		
	a	b	
0–30	0.359	0.250	0.300**
30–60	0.223	0.385	0.417**
60–90	0.084	0.519	0.564**

\*\* – statistically significant at a 99% probability ( $p \leq 0.01$ )

As note Wiltshire and du Preez (1994),  $\text{N-NH}_4^+$  concentration in soil increased depending on sample drying temperature, and it had almost no effect on nitrate levels. An increase in  $\text{N-NH}_4^+$  release from soil resulting from soil sample drying was also found by the USA researchers. In addition, on the basis of their findings it can be observed, that soil sample drying resulted in higher release of  $\text{N-NH}_4^+$  from the soil plough layer compared to deeper layers (Frye, Hutcheson, 1981).

The highest organic C and microorganism biomass is accumulated in the arable soil layer (Badalucco, Kuikman, 2001). The intensity of mineralization and nitrification of the organic matter depends not only on

moisture and temperature, but also on soil pH (Campbell et al., 1994; Kemmitt et al., 2006). However, the effect of pH on the activity of microorganisms is greatly reduced, when soil contains low amount of mobile Al and Mn (Curtin et al., 1998; Xiao et al., 2014). For the reasons mentioned above, it is difficult to evaluate the influence of pH on ammonification and nitrification processes in the soil from the agronomic point of view (Rousk et al., 2009). In addition, ammonia nitrogen in the soil is very mobile (Galloway et al., 2004).

Nevertheless, using the correlation equations presented in Table 3, it is possible to calculate  $\text{N-NH}_4^+$  concentrations in the field-moist samples theoretically according to the levels of these elements in the oven dried samples taken from the 0–30, 30–60 and 60–90 cm soil layers. Whereas the dependence is described by linear equations, the relationship derived between the levels of nitrogen in the field-moist and oven-dried soil samples can be used for conversion.

Soil nitrogen content ( $\text{N}_{\text{min}} = \text{N-NH}_4^+ + \text{N-NO}_3^-$ ) established in the oven dried soil samples taken from the 0–60 cm layer is evaluated according to the conditional groups of nitrogen (Staugaitis et al., 2007). It was therefore important to assess the dependence of  $\text{N-NH}_4^+$  and  $\text{N-NO}_3^-$  concentrations determined in the field-moist samples on the concentrations of these elements in the oven-dried samples of soils with different nitrogen content. The studies showed that with an increase of soil mineral nitrogen ( $\text{N}_{\text{min}}$ ) content at the 0–60 cm layer, the ratio between  $\text{N-NO}_3^-$  concentrations in the field-moist samples and the concentrations of this element in the oven-dried samples decreased (Table 4). At nitrogen content being  $<5.0$ ,  $5.1-10.0$  and  $>10.0$   $\text{mg kg}^{-1}$ , the ratio of  $\text{N-NO}_3^-$  concentrations in the oven-dried samples with the concentrations of this element in the field-moist samples was 1:1.33, 1:1.17 and 1:1.11, respectively. Although relatively higher  $\text{N-NO}_3^-$  concentrations were determined in the soils of lower ( $<5.0$  and  $5.1-10.0$   $\text{mg kg}^{-1}$ ) nitrogen content, in actual figures; however, more of those compounds were released from the samples of larger ( $>10.0$   $\text{mg kg}^{-1}$ ) nitrogen content soils during storage. At nitrogen content (in 0–60 cm layer) of  $<5.0$ ,  $5.1-10.0$  and  $>10.0$   $\text{mg kg}^{-1}$   $\text{N-NO}_3^-$  levels in the field-moist samples found were 0.68, 0.81 and 1.20  $\text{mg kg}^{-1}$  higher, respectively, compared to the content of this element in the oven-dried samples. The increase of  $\text{N-NO}_3^-$  levels in the field-moist samples was statistically significant compared to the oven-dried ones, except in the case of higher nitrogen content soils with  $\text{N}_{\text{min}} > 10.0$   $\text{mg kg}^{-1}$ .

With an increase of soil nitrogen content  $\text{N-NH}_4^+$  concentration in soil increased relatively less significantly, when compared to the variations in the levels of  $\text{N-NO}_3^-$  (Table 4). However, having dried the soil samples prior to carrying out chemical analyses,  $\text{N-NH}_4^+$  concentrations found in soil were statistically significantly higher, when compared to the levels of this element in the field-moist samples. At soil nitrogen content of  $<5.0$ ,  $5.1-10.0$  and  $>10.0$   $\text{mg kg}^{-1}$   $\text{N-NH}_4^+$  concentrations in the field-moist samples were 1.08, 1.21 and 1.11  $\text{mg kg}^{-1}$  higher, respectively, than those in the oven dried samples. Meanwhile, the ratio between  $\text{N-NH}_4^+$  concentrations in the field-moist samples and the concentrations of this element in the oven-dried samples of the soils with lower nitrogen content was found lower and that of the samples of higher nitrogen content soils – higher.

After the correlation-regression analysis of data, significant correlations between  $\text{N-NH}_4^+$  concentrations in the field-moist ( $y_1$ ,  $y_2$ ) and oven-dried ( $x_1$ ,  $x_2$ ) soil samples were determined (Table 5).

**Table 4.** Ammonium (N-NH<sub>4</sub><sup>+</sup>) and nitrate (N-NO<sub>3</sub><sup>-</sup>) nitrogen concentrations in oven-dried and field-moist samples of soils with different mineral nitrogen (N<sub>min</sub>) contents

N <sub>min</sub> concentration in 0–60 cm soil layer mg kg <sup>-1</sup>	N-NH <sub>4</sub> <sup>+</sup>		N-NO <sub>3</sub> <sup>-</sup>	
	oven-dried samples			
	field-moist samples			
	mg kg <sup>-1</sup>	ratio	mg kg <sup>-1</sup>	ratio
<5.0 (n = 176)	1.88** 0.80	1 0.42	2.03 2.71**	1 1.33
5.1–10.0 (n = 276)	2.16** 0.95	1 0.44	4.63 5.44**	1 1.17
>10.0 (n = 67)	2.24** 1.13	1 0.50	10.5 11.7	1 1.11

Note. \*\* – statistically significant differences between N-NH<sub>4</sub><sup>+</sup> or N-NO<sub>3</sub><sup>-</sup> concentrations determined in oven-dried and field-moist samples at a 99% probability ( $p \leq 0.01$ ).

**Table 5.** Relationship between ammonium nitrogen (N-NH<sub>4</sub><sup>+</sup>) concentrations in field-moist samples (y) and its concentration in oven-dried samples (x) of soils with different mineral nitrogen (N<sub>min</sub>) content

N <sub>min</sub> concentration in 0–60 cm soil layer mg kg <sup>-1</sup>	Parameters of equation $y = a + bx$		Correlation coefficient <i>r</i>
	a	b	
<5.0 (n=176)	0.036	0.404	0.392**
5.1–10.0 (n=276)	0.246	0.323	0.349**
>10.0 (n=67)	0.514	0.274	0.358**

\*\* – statistically significant at a 99% probability ( $p \leq 0.01$ )

The dependence of N-NH<sub>4</sub><sup>+</sup> levels in the field-moist samples (y<sub>2</sub>) on the concentration of this element in the oven-dried samples (x<sub>2</sub>) was low ( $r = 0.349–0.392$ ), although statistically significant, at a 99% probability (Table 4). These trends were observed in all soil nitrogen content groups (<5.0, 5.1–10.0 and >10.0 mg kg<sup>-1</sup>).

## Conclusions

1. Drying of soil samples in a thermostatic chamber at a temperature of 35°C for 12 hours resulted in significantly higher levels of ammonium nitrogen (N-NH<sub>4</sub><sup>+</sup>) compared to those of field-moist samples.

2. The nitrate nitrogen (N-NO<sub>3</sub><sup>-</sup>) concentrations in the field-moist samples, when stored at a 4°C temperature for three days, increased marginally compared to the oven-dried soil samples.

3. The N-NH<sub>4</sub><sup>+</sup> and N-NO<sub>3</sub><sup>-</sup> concentrations in both oven-dried and field-moist samples depended on the depth of sampling and soil nitrogen content.

4. The concentration ratios of N-NH<sub>4</sub><sup>+</sup> found between the oven-dried and field-moist samples at different (0–30, 30–60 and 60–90 cm) soil depths were 1:0.39, 1:0.52 and 1:0.58, respectively, and those of N-NO<sub>3</sub><sup>-</sup> – 1:1.20, 1:1.15 and 1:1.13.

5. At nitrogen content being <5.0, 5.1–10.0 and >10.0 mg kg<sup>-1</sup> in the 0–60 cm soil depth, the ratio of N-NH<sub>4</sub><sup>+</sup> concentrations in the oven-dried samples with the concentrations of this element in the field-moist samples was 1:0.42, 1:0.44 and 1:0.50, and that of N-NO<sub>3</sub><sup>-</sup> was 1:1.33, 1:1.17 and 1:1.11, respectively.

6. Weak or moderate, however, statistically significant correlations were observed between N-NH<sub>4</sub><sup>+</sup> concentrations in the field-moist and oven-dried soil samples.

7. In order to perform faster chemical analyses of soil mineral nitrogen (N<sub>min</sub>) for making agricultural plant fertilisation plans, it is necessary to carry out analyses on oven-dried soil samples, and using the linear correlation

equations, to convert the results of N-NH<sub>4</sub><sup>+</sup> analyses into field-moisture data.

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## Mineralinis azotas Lietuvos žemės ūkio naudmenų dirvožemiuose: orasausių ir lauko drėgnio ėminių palyginimas

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### Santrauka

Mineralinis azotas ( $\text{N}_{\text{min}} = \text{N-NH}_4^+ + \text{N-NO}_3^-$ ) tirtas 2011–2014 m. penkiose Lietuvos vietovėse, orasausius ir lauko drėgnio dirvožemio ėminius paėmus iš skirtingos granulometrinės sudėties (smėlio, smėlingo priemolio, priemolio ir molio) ir skirtingo azotingumo dirvožemių. Ėminiai buvo imti pavasarį (kovo pabaigoje – balandžio pradžioje) ir rudenį (spalio pabaigoje – lapkričio pradžioje), iš dirvožemio 0–30, 30–60 bei 60–90 cm ir 0–60 cm sluoksnių. Tyrimo tikslas – nustatyti dirvožemio ėminių paruošimo skirtingų metodų įtaką amoniakinio ( $\text{N-NH}_4^+$ ) ir nitratinio ( $\text{N-NO}_3^-$ ) azoto koncentracijoms orasausiuose bei lauko drėgnio ėminiuose ir įvertinti gautų rezultatų tinkamumą sudarant žemės ūkio augalų tręšimo planus.

Tyrimo duomenimis, dirvožemio mėginius termostate džiovinant 12 valandų 35 °C temperatūroje, amoniakinio azoto nustatoma reikšmingai daugiau, palyginus su lauko drėgnio mėginiais. Nitratinio azoto koncentracija lauko drėgnio mėginiuose, juos sandėliuojant 3 paras 4 °C temperatūroje, padidėjo nežymiai, palyginus su orasausiais. Lauko drėgnio ir orasausiuose dirvožemio mėginiuose tarp amoniakinio azoto koncentracijų nustatyti silpni arba vidutinio stiprumo, tačiau esminiai koreliaciniai ryšiai. Sudarant žemės ūkio augalų tręšimo planus ir siekiant greičiau atlikti dirvožemyje esančio mineralinio azoto chemines analizes, reikia analizuoti orasausius dirvožemio mėginius, o amoniakinio azoto analizių rezultatus pagal linijinės koreliacijos lygtis perskaiciuoti į lauko drėgnį.

Reikšminiai žodžiai: amoniakinis ir nitratinis azotas, ėminių paruošimas analizėms.