Evaluation of measurement uncertainty by sampling on the example of determination of mobile phosphorus compound in the soil In accordance with the requirements of ISO / IEC 17025:2017

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Introduction

Uncertainty of measurement is the most important general parameter of characteristic of measurement quality and has a great influence on decisions that is made based on measurement results. Methods for estimating the uncertainty of the sampling process are insufficient. In order to make the right decision is based on the measurement results, it is necessary to take the uncertainty into account, which is associated with the sampling process when assessing is uncertainty. We developed an estimate of the total uncertainty of the measurement by sampling, sampling, and analyzing the uncertainty of the individual components by using a model approach and an example approach to determine the mobile phosphorus compound in the soil.

Methodology

Sampling scheme was drawn up: 20 samples were taken at about per hectare from a depth of 30 cm using a soil drill; Factors which are affecting the measurement were identified and may be related to sampling tools, sampling error, soil moisture, or soil sample which are the loss from the sampling device. Because of that these factors are difficult to determine individually, they are generally referred to as the "depth effect"; shredding and distribution during sample preparation reduce the amount of soil sample. We have a cause-and-effect diagram of the measurement process (Figure **1**). Samples were taken by conical and quartering methods, air dried and drilled through a hole <2 mm in diameter. Determination of the phosphorus mobile compound was performed by the Oniani method (modified by Cinao).

Results and discussion

The plot was divided into nine squares (A, B, C x 1, 2, 3) and five squares ("crosswise" across the plot), which are selected with five separate samples result of the measurement was determined by the arithmetic mean of the results of 5 separate samples. The concentration of the mobile compounds phosphorus measured in the five squares is: A1 - 500 mln^{-1}; A3 – 498 mln^{-1}; B2 – 502 mln^{-1}; C1 – 500 mln^{-1}; C3 – 498 mln^{-1}. Xscr = X ana – 500 mln^{-1}; 2 mln^{-1} (0,4%). Standard deviation between measurement values (ssqr between samples have taken from a single quadratic. $u_{b\text{-}loc} = s_{sqr}/\sqrt{n}_{b\text{-}loc}$=0,179% (1). A special experiment was conducted to detect the total effect of the "depth effect"'s factors. Samples from 35 cm deep were taken in five "test squ Segments 25-30 cm and 30-35 cm were separated from them and then the selected segments were combined from different squares. The uncertainty c by the "depth effect" was estimated by the content of phosphorus below and above the nominal depth in the soil layers (c_1, c_2). In particular, the phosp content is: c_1 (25-30 см) - 350 mln^{-1}; c_2 (30-35 см) – 335 mln^{-1}. The upper and lower limits were estimated: x_+ - 485 mln^{-1}; x_- - 517 mln^{-1}; $\Delta_{x\_}$ 32 Standard uncertainty u_{depth} = 9,25 (Table 2). When we are distributing the samples, we follow the initial samples 2 to 7 times by the method of conjug and quartering, the mean standard deviation is - 1,2 (Table 3), u_{dry} =0,6%. The standard uncertainty of input type A is $u_A(x_i)$ tp (v)/kp=1,085. Determ input values: sampling between sampling sites; sampling strategy; depth; sample splitting; drying; The standard uncertainty of the measuring utensils, tools, reagents and the type of probability distribution: uncertainty budget (Figure.2; Table 4). Extended uncertainty of the mass fraction of the m compounds of phosphorus in the soil U = 20,33.

The result of the measurement is 500 ± 20.33 mln^{-1}.

Table 1. Measured concentration of mobile compounds of the phosphorus in five squares

Square	Concentration of mobile compounds of the phosphorus, mln^{-1}
A1	500
A3	498
B2	502
C1	500
C3	498
—	500
Xscr = X ana	
ssqr	2
	(0,4%)
ub-loc	0,179%

Table 2. Depth experiments

	P, mln^{-1}
c_1 (25-30 cm)	350
c_2 (30-35 cm)	335
x_-	517
x_+	485
$\Delta_{x\_}$	32
u_{depth}	9,25

Figure 1 Cause-and-effect diagram for soil sampling on arable land (Rw is within-laboratory reproducibility)

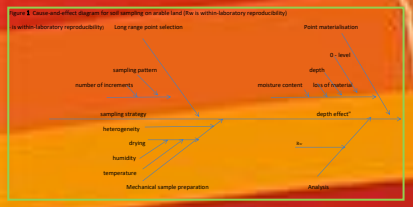

Figure. 2 Uncertainties in KH_2PO_4 Solution preparation

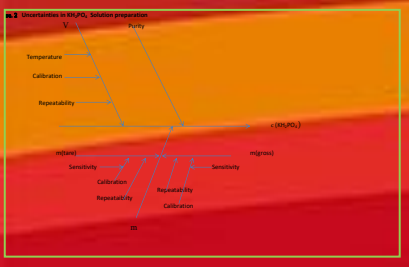

Table 4. Uncertainty Budget

Input quantities	Estimate of input quantities	Standard uncertainty of input quantity	Type of distribution of probability
The distance between the sampling points		0,18	Normal
Sampling strategy		0,5	Rectangular
Depth		9,2	Normal
Division of sample		1,2	Normal
Drying		0,6	Normal
Quality of purity		0,0046	Rectangular
Mass		0,00006	Rectangular
Volume		0,1	Rectangular
Analysis		Type A total standard uncertainty $u_{\sum A}(X_i) = 1,085$	Normal
200 ml measuring flask	200ml	U_{200f}= ±0,091	Rectangular
50 ml measuring cylinder	50 ml	U_{50p}= ± 0,152	Rectangular
Error of 50 ml measuring cylinder count		$u_{c\ 50m.c.}$ = ±0,303 ml	Rectangular
2 ml pipette	2 ml	u_{2p}= ±0,012	Rectangular
Error of 2 ml pipette count		$u_{c\ 2p}$ = ±0,006 ml	Rectangular
Standard uncertainty of measuring cylinder and pipette volume u(V)	50 ml 2 ml	u(V)=0,11	Rectangular
The degree of purity of ascorbic acid		0,0013	Rectangular
The degree of purity of sulfuric acid		0,0006	Rectangular
The degree of purity of Antimony potassium tartrate		0,0053	Rectangular
The degree of purity of ammonium molybdate		0,0012	Rectangular
Photoelectrocolorimeter КФК-3-01 УХЛ 4.2 №GMC-CC0-0669/5-20		$u_{kpk\text{-}3}$ =0,0014	Normal
Scale laratotorial ISO LAB	(0-320) g	$u_{c\ scale\ 320}$ =0,0029 g	Normal

Table 3. Relative standard deviations duplicate split samples and the mean standard deviations for both analytes

Square	P, mln
A1	0
A3	2
B2	2
C1	0
C3	2
–	1,2
S split	

Conclusion

The result obtained m
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