



GLOSOLAN
Proficiency Test
for Eurasia
2023



PHOSAGRO[®]

Results of GLOSOLAN PT for Eurasia 2023. Soil organic carbon. Tyurin and Walkley-Black methods

Webinar
29 October 2024

Shamrikova E.
Chair of RUSOLAN
shamrikovaelena@yandex.ru



Main authors:

Boris Kondratenok – Deputy Director in Science, Candidate of Chemistry

Elena Kyzyurova – Lead chemical engineer

Elena Lapteva – Head of the Department of Soil Science, Candidate of Biology

Evgenia Tumanova – Lead chemical engineer

Evgenia Vanchikova – Candidate of Chemistry

Natalia Bondarenko – Engineer of the 1 category

Olga Ostanina – Lead chemical engineer

Svetlana Kostrova – Head of the ecoanalytical laboratory

Tatyana Zonova – Lead chemical engineer

Yulia Bobrova – Lead chemical engineer

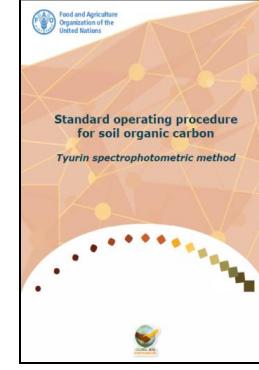
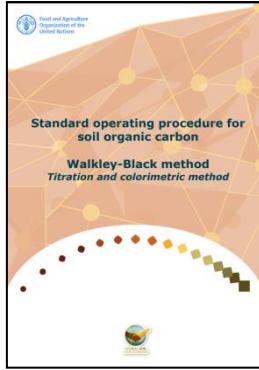
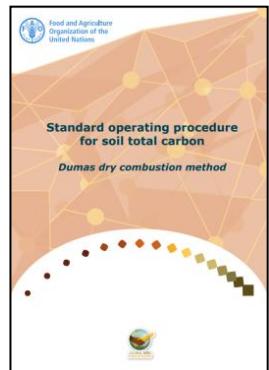


Tested methods

- Dry combustion on the analyzer (**DC**),
- Walkley-Black's method (**W-B**),
- Tyurin's method (**T**) – %SOC = 0.17-8.7,
- Loss-on-ignition method (**LOI**)

SOPs of GLOSOLAN

SOP of GLOSOLAN in progress



Aim –

to identify factors influencing the quality of SOC measurements using the Tyurin and Walkley-Black methods

Misses

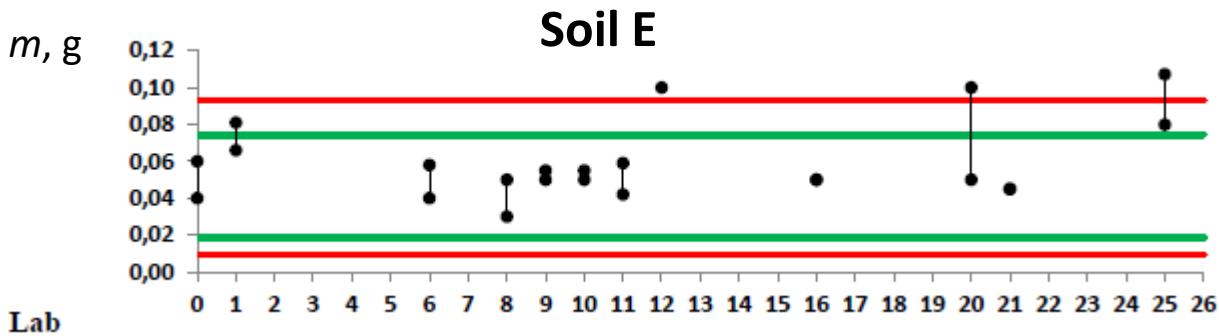
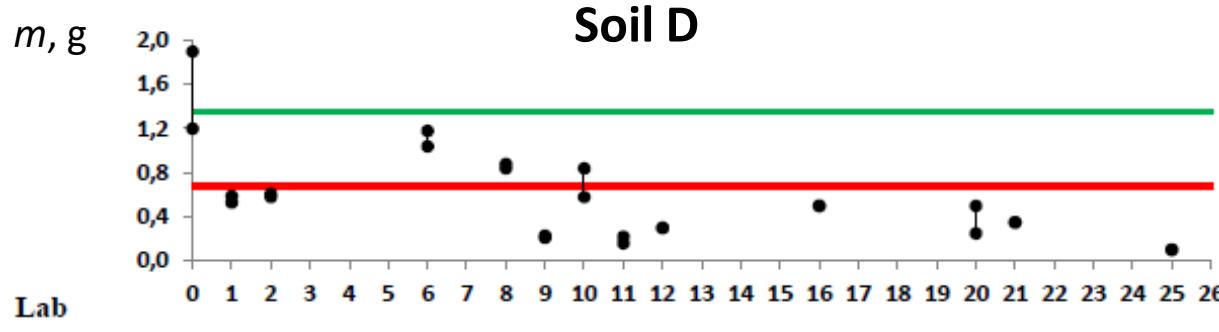
Control soils	%SOC	Misses
Soil A = Soil F = Soil I	6.44	no
Soil B = Soil G = Soil H	2.32	
Soil E	7.87	
Soil C	0.17	29 of 54
Soil D	0.1	



Lab code	Methods									
	T				W-B					
	Soils									
	A F I	B G H	C	D	E	A F I	B G H	C	D	E
Lab_1										
Lab_2										
Lab_3										
Lab_4	-	-	-	-	-	-	-	-	-	-
Lab_5										
Lab_6										
Lab_7	-	-	-	-	-	-	-	-	-	-
Lab_8										
Lab_9										
Lab_10										
Lab_11										
Lab_12										
Lab_13										
Lab_14	-	-	-	-	-	-	-	-	-	-
Lab_15										
Lab_16										
Lab_17										
Lab_18										
Lab_19	-	-	-	-	-					
Lab_20										
Lab_21										
Lab_22										
Lab_23										
Lab_24										
Lab_25										

Factors Affecting the Quality of Results

1. Soil mass. Tyurin's method

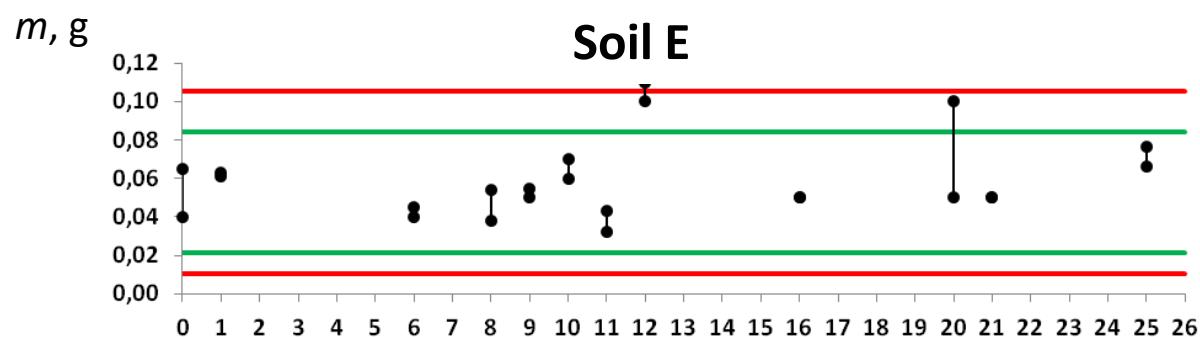
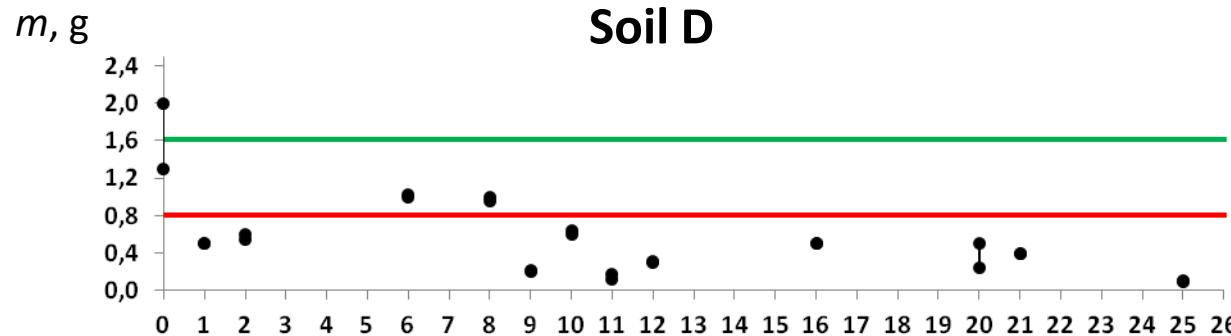


- $A = 0,05\text{--}0,5$ – Working range of the Calibration curve
- $A = 0,1\text{--}0,4$ – Optimal range of the Calibration curve

Recommended weighed of soils (SOP)

Soil color	SOC	Weight of soil
		<i>m</i>
	%	<i>mg</i>
Whitish	From 0.17 to 0.6 incl.	From 500 to 900
Light gray, light brown	From 0.6 to 1.2 incl.	From 200 to 500
Gray, brown	From 1.2 to 3.0 incl.	From 100 to 200
Dark gray, brown	From 3.0 to 6.0 incl.	From 60 to 100
Dark brown, black	From 6.0 to 8.7 incl.	From 30 to 60

1. Soil mass. Walkley-Black's method

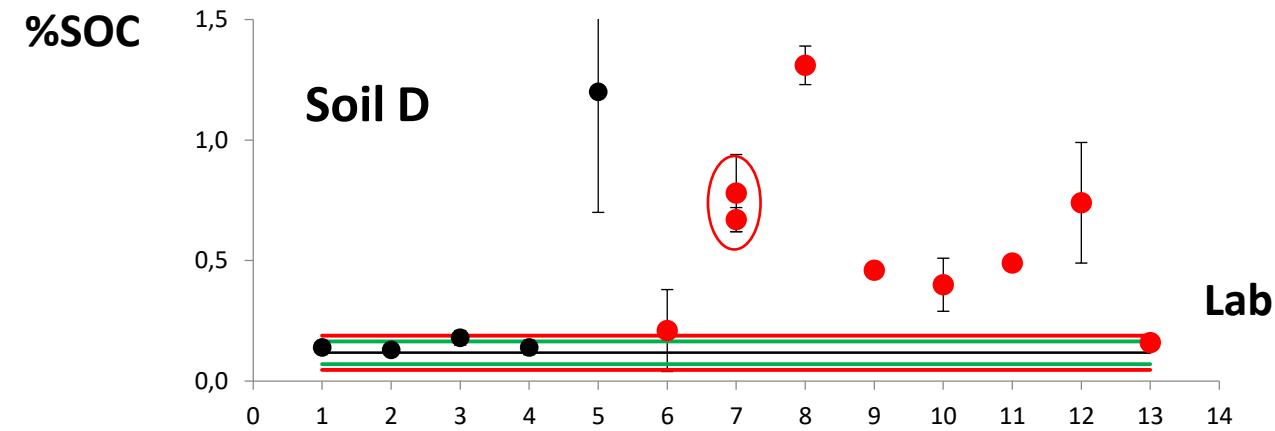
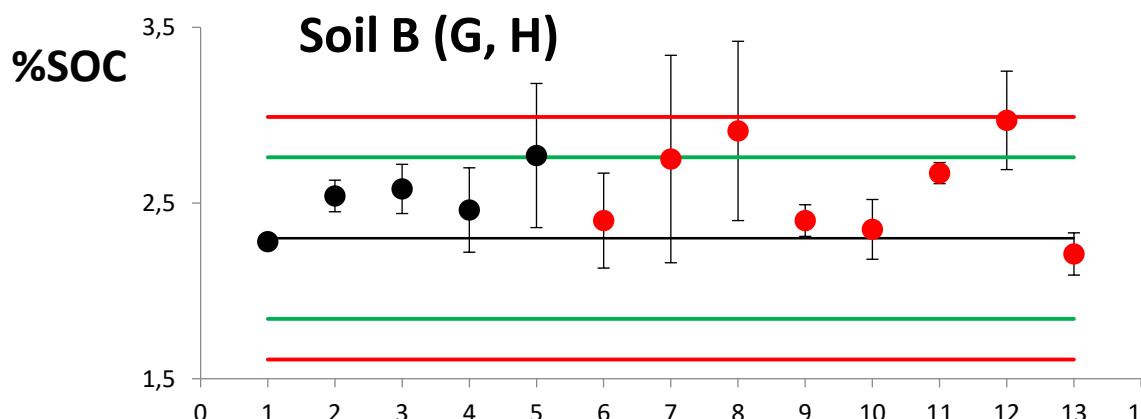
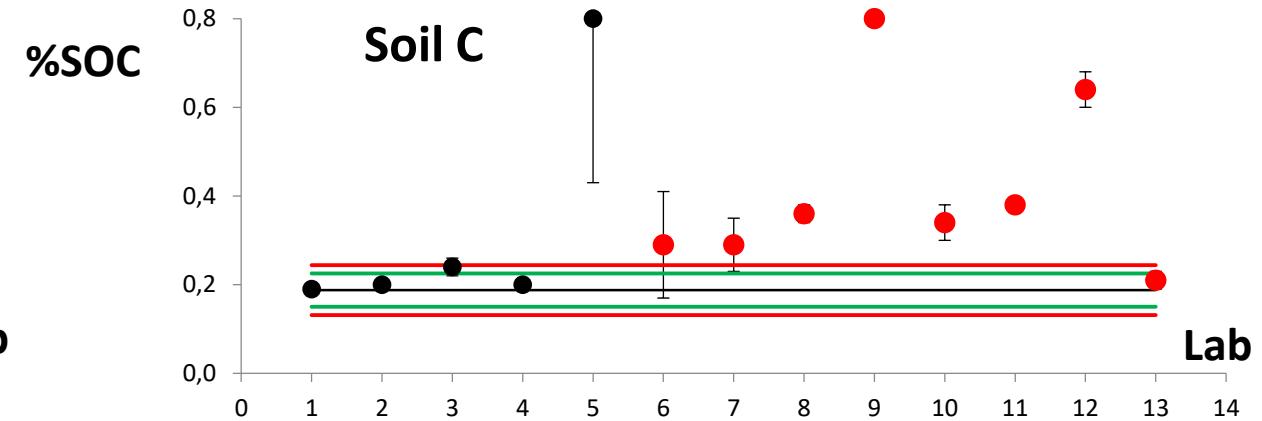
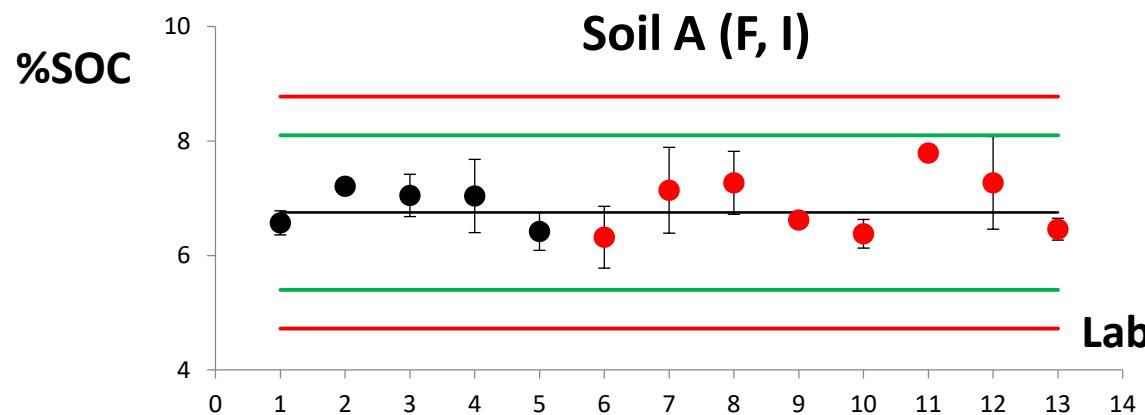


— $A = 0,05\text{--}0,5$ – Working range of the Calibration curve
— $A = 0,1\text{--}0,4$ – Optimal range of the Calibration curve

Recommended weighed of soils (SOP)

Weight, g	OC, %	Color
0,1	>2	black, dark gray, dark brown
0,25	≤2	brown - dark brown, gray - dark gray
0,5	<0,6	Brown

2. Separation of solid and liquid phases after oxidation of the SOC. Tyurin's method



● – centrifugation (SOP)
● – settling

Soils. Granulometric composition (Kachinsky's method)

Soils	H ₂ O	Acid soluble compounds	1–0.25	0.25–0.05	0.05–0.01	0.01–0.005	0.005–0.001	< 0.001
			mm					
			%					
Soil A(F, I)	2.80	5.08	5.01±0.02	13.4±1.5	50.6±4.2	4.0±0.9	9.5±0.5	14.8±0.5
Soil B(G, H)	1.52	1.61	2.14±0.20	11.3±0.9	64.3±0.5	3.9±0.9	4.9±0.2	15.1±1.9
Soil C	0.24	0.25	10.7±1.2	78.5±1.4	5.8±1.4	0.4±0.3	0.4±0.3	4.1±0.7
Soil D	2.38	1.40	8.5±0.4	29.8±0.9	20.4±2.5	4.3±1.0	11±4	25.0±2.1
Soil E	5.20	3.26	31.9±1.3	9.1±2.0	13.9±1.9	8.5±1.0	21.6±0.5	16.7±0.2

2. Separation of solid and liquid phases after oxidation of the SOC. Tyurin's method

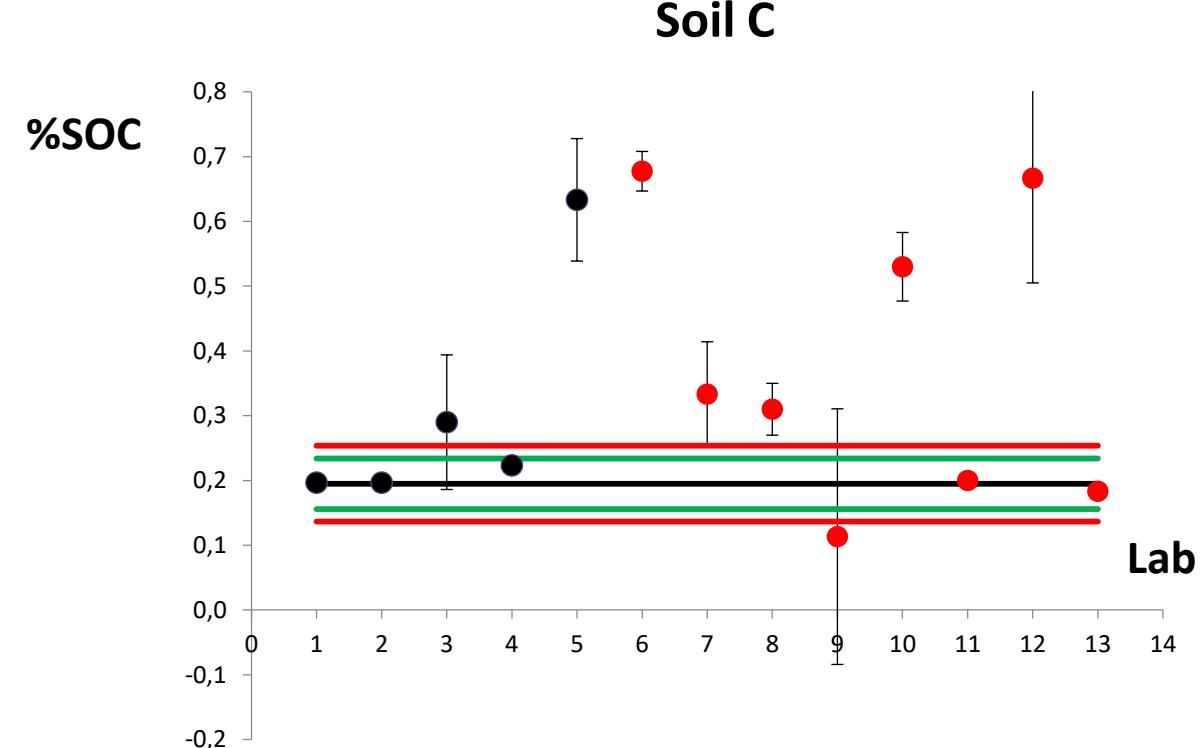
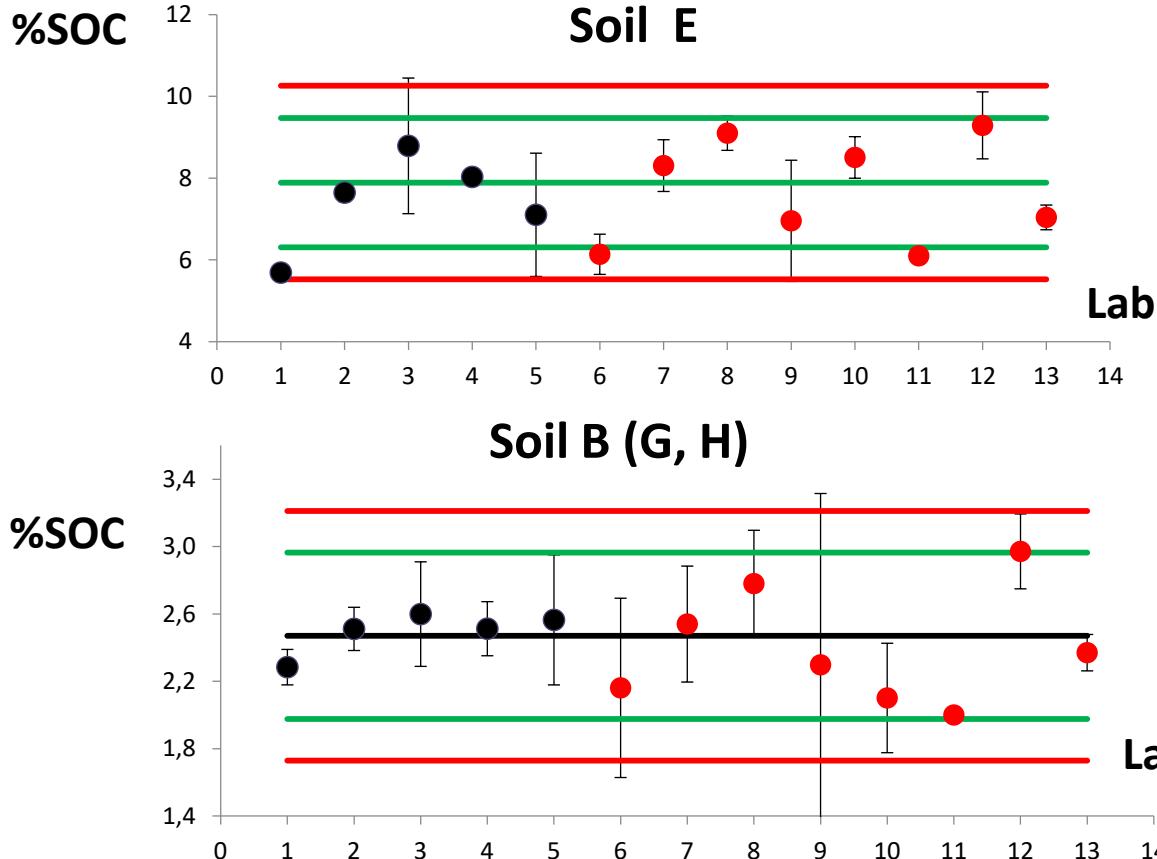
Settling / Centrifugation ???



Soil D (Lab 9)				
%SOC ₁	%SOC ₂	%SOC ₃	<i>U, %</i>	Settling
0.66	0.65	0.70	540	experiment 1
0.69	0.81	0.84	640	experiment 2 (after 9 days)

U – offset from control value

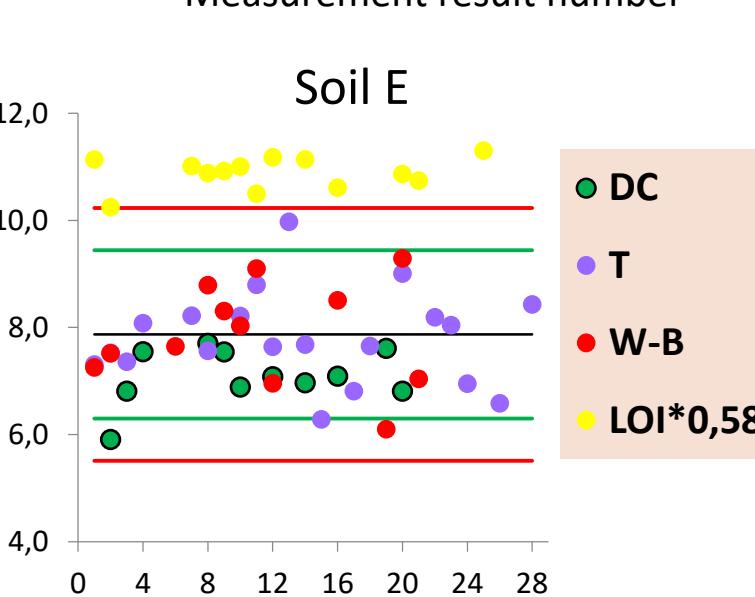
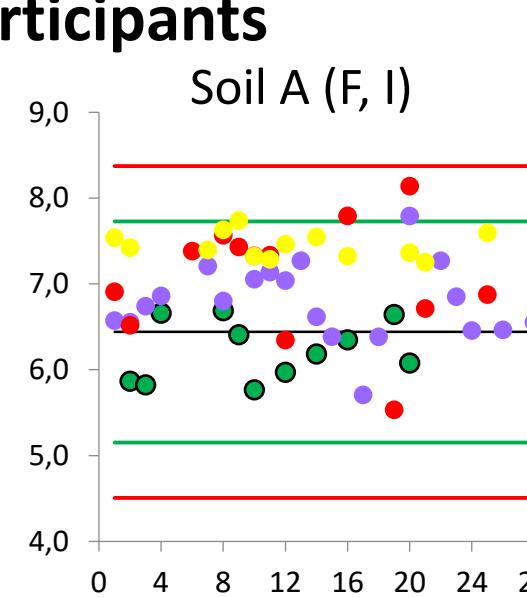
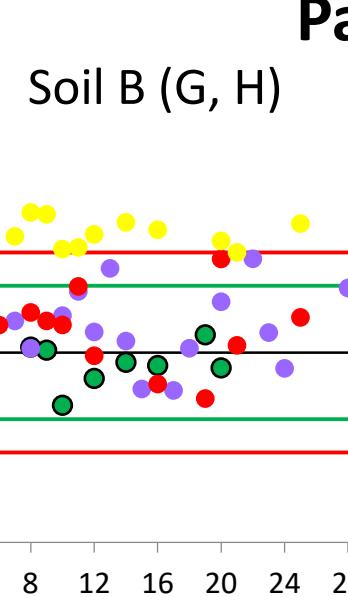
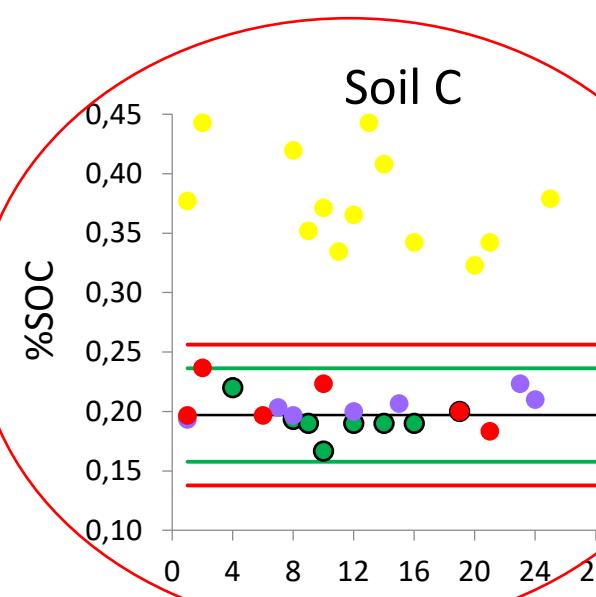
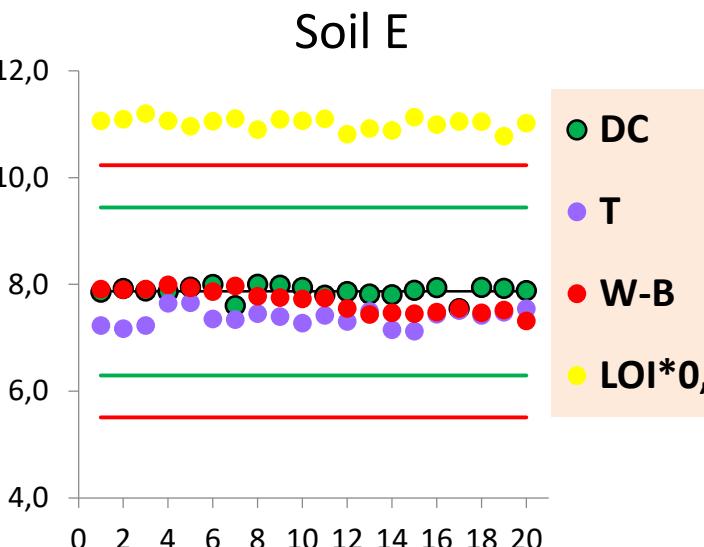
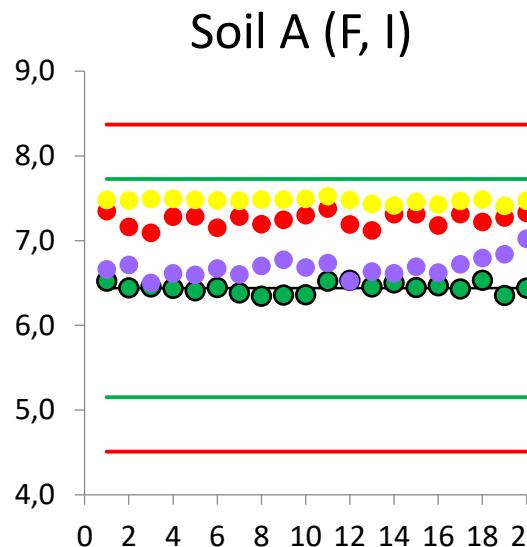
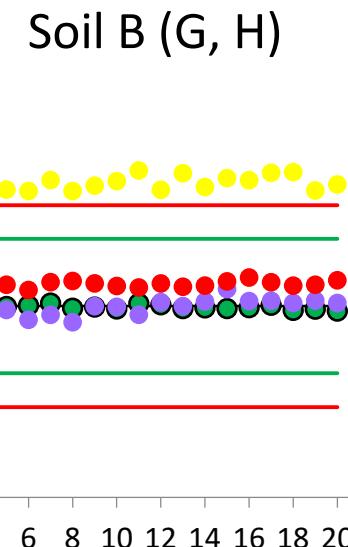
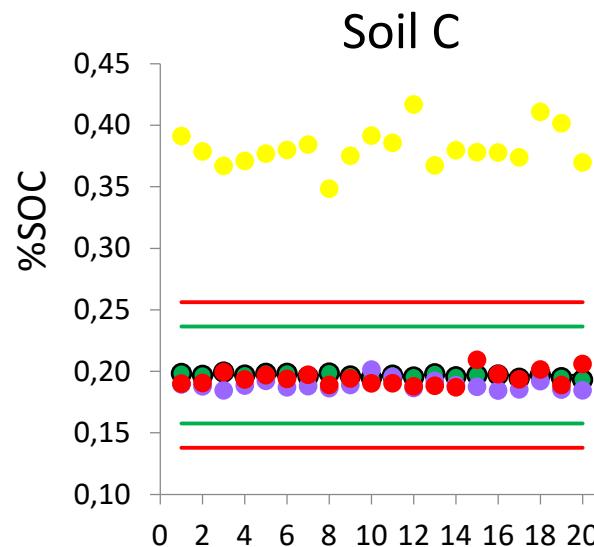
2. Separation of solid and liquid phases after oxidation of the SOC. Walkley-Black's method



● – centrifugation (SOP)
● – settling

NatRefLab (RUSOLAN)

%SOM = %LOI, %SOC = %LOI * 0,58



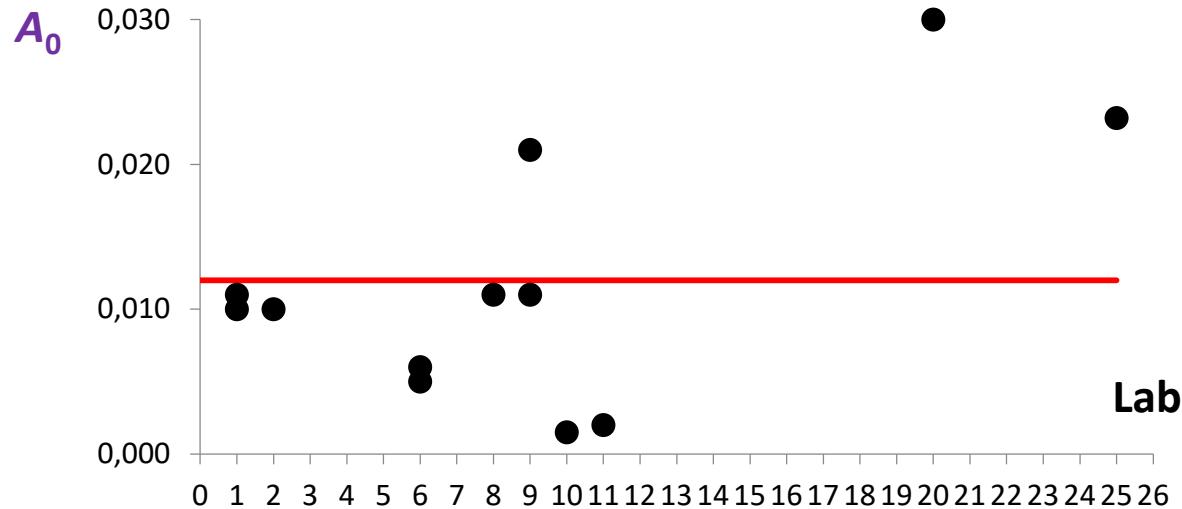
Participants

DC
T
W-B
LOI*0,58

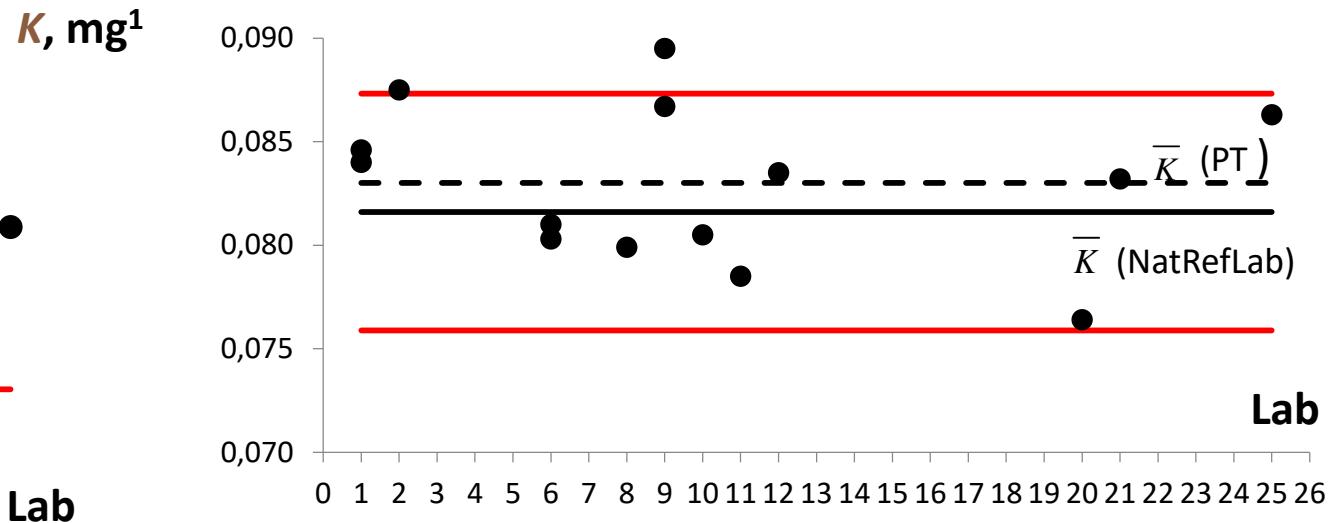
DC
T
W-B
LOI*0,58

3. Calibration curve (CC) coefficients ($A = Km(C_{org}) + A_0$, R^2)

Tyurin's method



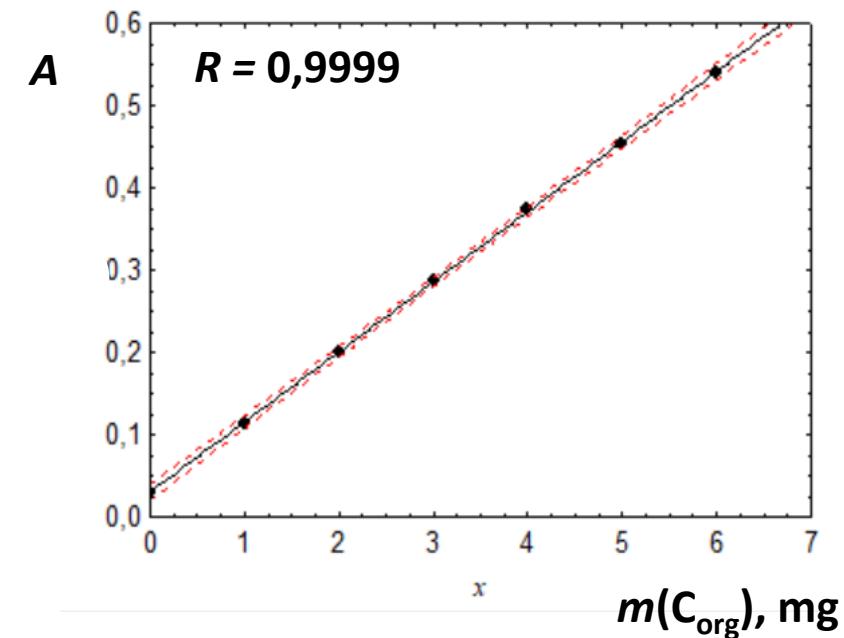
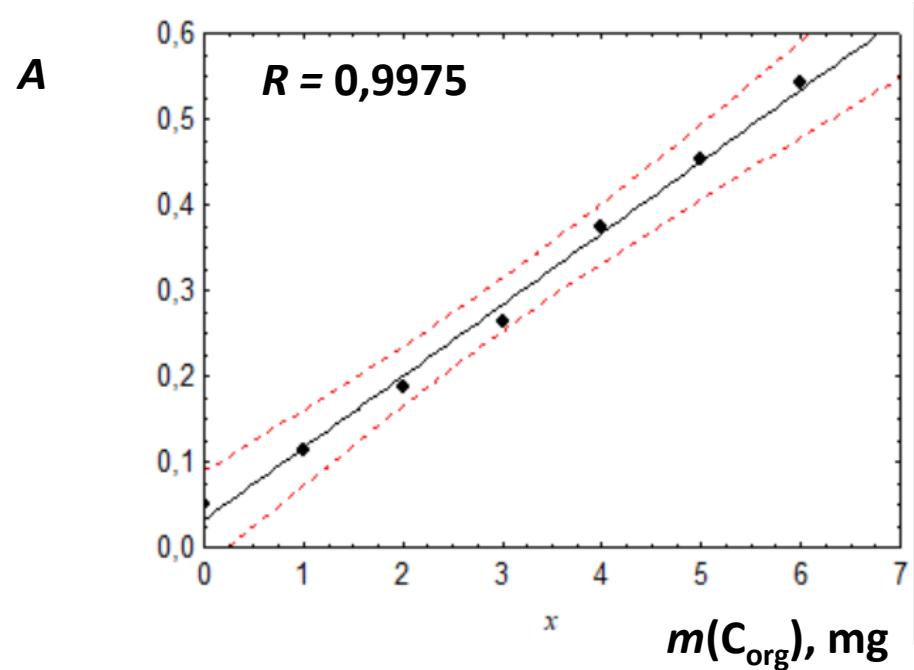
- cleanliness of cuvettes,
- correct preparation of blank solution,
- correct preparation of reducing agent solution



	Characteristics of CC			Data for the stability map of the CC coefficient				
	A_0	K , mg ⁻¹	R		K , mg ⁻¹	\bar{K}	$\bar{K} + 2S(K)$	$\bar{K} - 2S(K)$
1	0,011	0,0819	0,9996	1	0,0819	0,0828	0,0851	0,0804
2	0,009	0,0841	1,0000	2	0,0841	0,0828	0,0851	0,0804
3	0,011	0,0845	0,9999	3	0,0845	0,0828	0,0851	0,0804
4	0,021	0,0831	0,9999	4	0,0831	0,0828	0,0851	0,0804
5	0,022	0,0837	0,9997	5	0,0837	0,0828	0,0851	0,0804
6	0,016	0,0834	0,9998	6	0,0834	0,0828	0,0851	0,0804
7	0,007	0,0818	0,9997	7	0,0818	0,0828	0,0851	0,0804
8	0,005	0,0819	0,9998	8	0,0819	0,0828	0,0851	0,0804
9	0,008	0,0826	0,9999	9	0,0826	0,0828	0,0851	0,0804
10	0,006	0,0808	0,9995	10	0,0808	0,0828	0,0851	0,0804

3. Calibration curve (CC) coefficients ($A = Km(C_{\text{org}}) + A_0$, R^2)

Tyurin's method



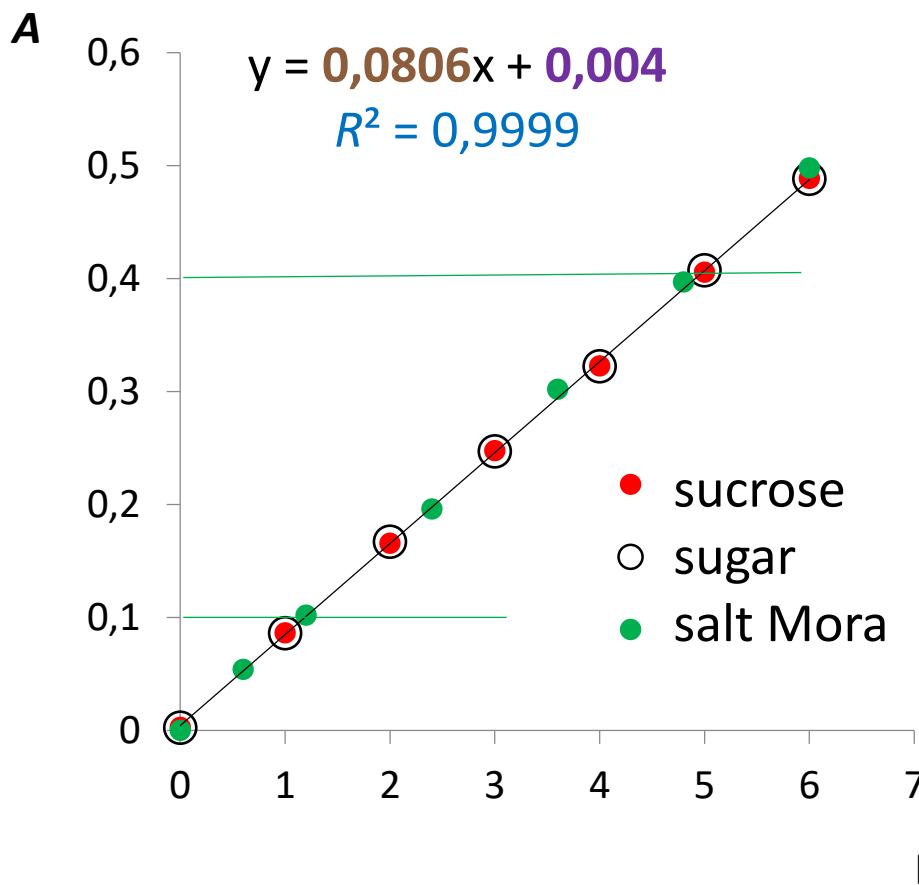
accuracy of preparation of each calibration solution

3. Calibration curve (CC) coefficients ($A = Km(C_{\text{org}}) + A_0$, R^2)

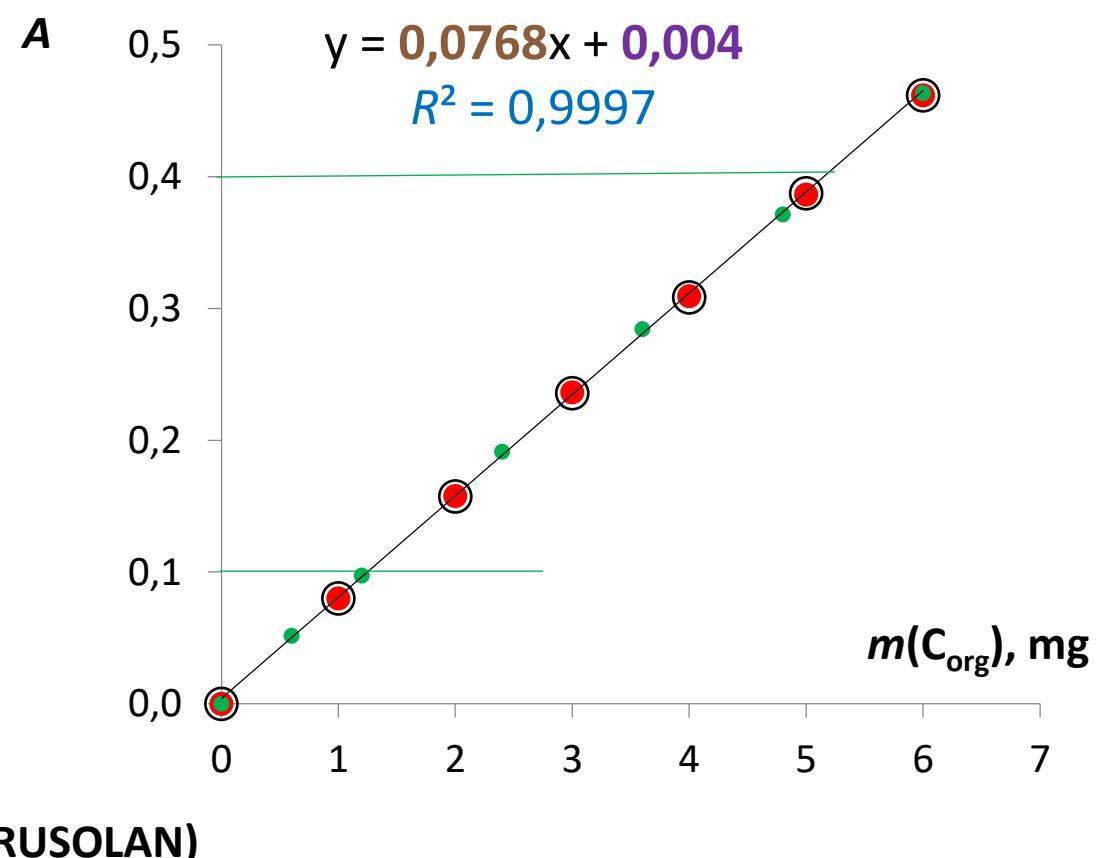
SOP: Working range of the Calibration curve – $A = 0,05–0,5$;

Optimal range of the Calibration curve – $A = 0,1–0,4$

Tyurin's method



Walkley-Black's method



4. Correction factors (modifications of Tyurin's method)



The coefficient $f = 1.15$ was applied to all results.

	Soil A (F, I)	Soil B (G, H)	Soil C	Soil D	Soil E
Tyurin's method					
Experimental coefficient f	1,10	1,16	1,20	1,28	1,19
Walkley-Black's method					
Experimental coefficient f	1,16	1,22	1,31	1,49	1,30

5. Problems with calculating (*rounding*) results

	Results			Precision	
	%SOC ₁	%SOC ₂	%SOC ₃	U_{abs}	U_{rel}
Excel	0,207	0,207	0,253	0,0531	23,8904
when using , the "dropped" numbers are taken into account in further calculations					
Excel	0,21	0,21	0,25	0,05	24
when re-transferring numbers to Excel after Word, the “discarded” numbers are not saved					
Excel	0,21	0,21	0,25	0,05	21

How to get quality results?

Walkley-Black * 1,3 = Tyurin * 1,15 = Dry Combustion ($P = 0,95$)

1. Strict adherence to protocol (centrifugation, conversion factor)
2. Selection of the optimal soil mass (Absorbance $A = 0,1-0,4$)
3. Control of calibration curve coefficients ($A = Km(C_{org}) + A_0, R$)



<https://ib.komisc.ru/rusolan/>

Thank you for attention!

