

Food and Agriculture Organization of the United Nations

Managing salt-affected soils for sustainable future

2nd Meeting of the International Network of Salt-Affected Soils (INSAS)

How to participate to international proficiency tests?

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> Hybrid meeting Tashkent/Nukus, Uzbekistan May 22-26, 2023



International Network of Salt-Affected Soils

Introduction

- *Soil* is a vital part of the natural environment and a resource that provides 95% of the world's population with food.
 - Agricultural soils are a strategic resource for guarantee food security.
- **Soils** provide living space, as well as perform ecosystem functions necessary to provide the inhabitants of the planet with clean water, climate regulation, and biodiversity conservation





Food Security

Sovereignt

work of Salt-Affected Soils (INSAS) | Tashkent/Nukus, Uzbekistan | May 22-26, 2023

Soil parameters



chemical;
physical;
biological



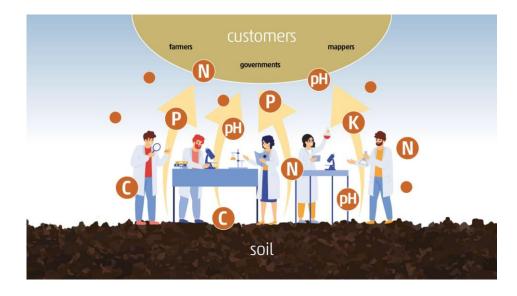
Soil parameters



chemical;
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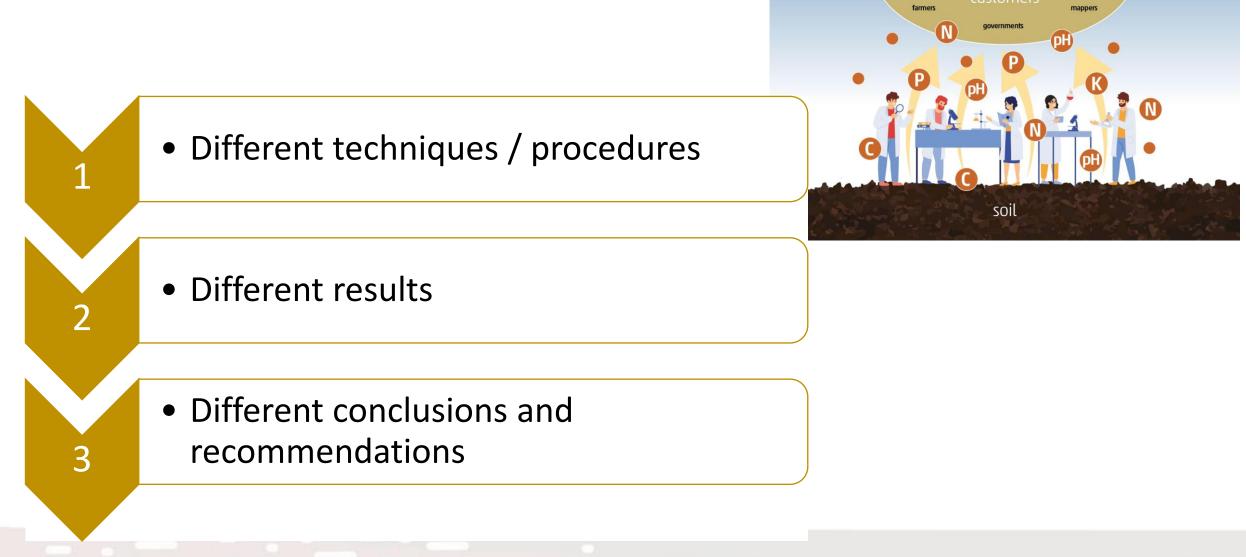
Information and methodological support for measuring parameters / indicators of soil quality and health





- the result of geographic isolation;
 - historical circumstances;
 - technical and economic limitations;
 - method suitability for local soils and calibrations

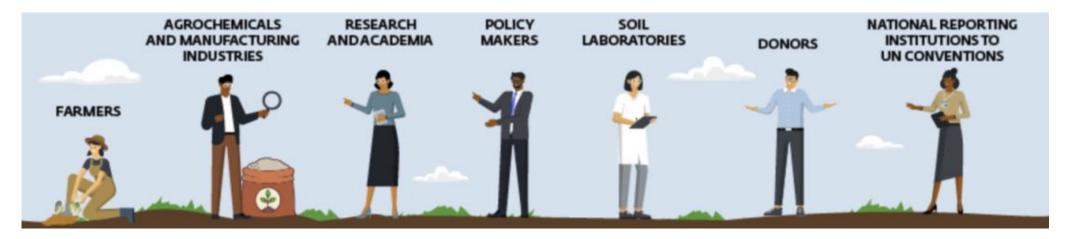






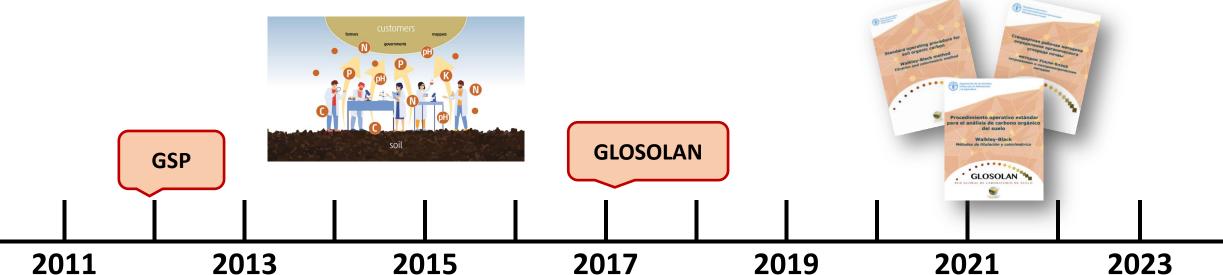
Adopt a common SOP for each soil parameter in all laboratories in the world

Soils: if you cannot measure it, you cannot manage it



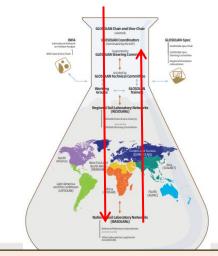


www.fao.org/global-soil-partnership/glosolan/



Already published:

- 1 on sample pre-treatment;
- 17 on soil chemical parameters (7 more ongoing);
- 1 on soil physical parameter (5 more ongoing);
- 1 on soil biological parameter (6 more ongoing)



960 laboratories in the world



Global Soil Laboratory Network (GLOSOLAN)

Major areas of work:

- Harmonization of Standard Operation Procedures (SOPs)
- Training on the implementation of GLOSOLAN SOPs
- Training on safety and health
- Execution of external quality control (proficiency testing)
- Training on the execution of internal quality control
 - Training on equipment use, maintenance and purchasing
 - Establishment of a donation/bartering system
 - Spectroscopy









Almost 9000 views in one year!

www.fao.org/global-soil-partnership/glosolan/

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External guality control (also called "proficiency testing" or "inter-laboratory comparison") is a periodic ass of the performance of individual laboratories and groups of laboratories. The assessment is done by an independent sting body like GLOSOLAN through the distribution of typical materials for unsupervised analysis by the participant roficiency testing is used as a tool to asses and enhance standards of the analysis, and assist in the standardisation o soil analytical methods across laboratories leading to more reliable and interoperable soil data. Laboratories can use results obtained in the PT to identify areas where improvements may

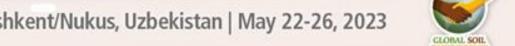


soil sample for proficiency testing n Soil and Plant Laboratories. (FAO AR I EN LES | FR | RU | ZH | TH Soils Bulletin - 74

www.fao.org/global-soil-partnership/glosolan/







Tested methods



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

Standard soil samples (9): A, B, C, D, E, F, G, H, I



Tested methods



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- Dry combustion on the analyzer (DC), Why SOC?
- Walkley-Black's method (WB)
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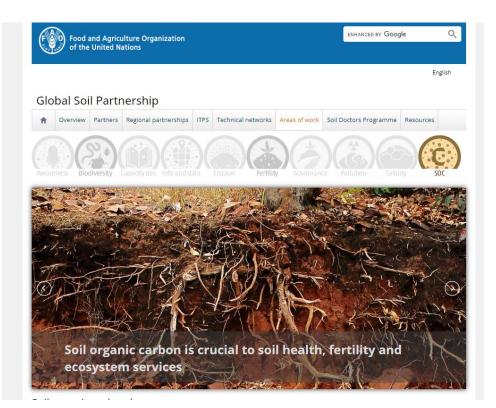


SOC is one of the most important components of soils.

In the age of global changes in the environment, monitoring SOC is of outmost importance.

Under the Global Soil Partnership initiative of the Food and Agriculture Organization of the United Nations (FAO), the availability of nonharmonized data is one of the reasons for the low accuracy of the global SOC map (*Peralta et al., 2022*).

This is especially true for regions such as Eurasia where data are sparse.







Measurement steps

Tyurin method (Colorimetric Method)

- 20g K₂Cr₂O₇ + 0,5L H₂O, c(K₂Cr₂O₇)= 0,136 M
- "Chromium mixture": $1V K_2Cr_2O_7+1V H_2SO_4_{conc}$ $c(K_2Cr_2O_7)=0,068 M$
- Soil+10 mL "Chromium mixture" (5 mL+5 mL)
- added to the sample $n(K_2Cr_2O_7)=0,68$ mmol
- Heating in a water bath (t = 100 °C for 60 min)
- +15 mL H₂O
- Centrifugation 6000 rpm for 10 min
- Measure the optical density λ = 590 nm
- Calculation of %Corg

Walkley-Black method (Colorimetric Method, GLOSOLAN)

- **50 g** $K_2Cr_2O_7$ +0,5L H_2O , $c(K_2Cr_2O_7)$ = 0,34 M
- Soil+ 2 mL $K_2Cr_2O_7$ +5 mL $H_2SO_{4 \text{ conc}}$ = 7 mL added to the sample $n(K_2Cr_2O_7)$ = 0,68 mmol
- Standing for 30 min
- +20 mL H₂O
- Standing for 24 hours (without external heating)
- Measure the optical density λ = 590 nm
- Calculation of %Corg

oxidation correction factor f = 1,0 - traditionally

oxidation correction factor *f* = 1,3

Tyurin I.V. New modification of the volumetric method for determining humus using chromic acid // Pochvovedenie, 1931. No. 6. P. 36-47.

Walkley A., Black I.A. An examination of the Degtjareff method for determining soil organic matter, and a proposed modification of the chromic acid titration method // Soil Sci., 1934. V. 37. P. 29-38.

$[C]^{0} + 2 Cr_{2}O_{7}^{2} + 16 H^{+} = 3 CO_{2} + 4 Cr^{3+} + 8 H_{2}O$





Measurement steps

Tyurin method

(Colorimetric Method)

Walkley-Black method (Colorimetric Method, GLOSOLAN)

- 20g K₂Cr₂O ! In the Walkley-Black method, the amount of K₂Cr₂O₇ and H₂SO₄ is equal to the same
- "Chromium characteristics as in the Tyurin method, but the concentration of these components of $c(K_2Cr_2O_7)=0$, the mixture is 1,5 times higher.
- Soil+10 mL chromium mature (5 mL + 5 mL) added to the sample $n(K_2Cr_2O_7)=$! Heating of the reaction mixture occurs due to the - Heating in a water bath (t = 10 exothermic effect that occurs when a concentrated
- Heating in a water bath (t = 10 exothermic effect that occurs when a concentrated
 +15 mL H₂O
 - solution of H₂SO₄ is mixed with distilled water.

xternal heating)

- Centrifugation 6000 rpm for 10
- Measure the optical density λ = 590 nm
- Calculation of %Corg

oxidation correction factor f = 1,0 - traditionally

Tyurin I.V. New modification of the volumetric method for determining humus using chromic acid // Pochvovedenie, 1931. No. 6. P. 36-47.

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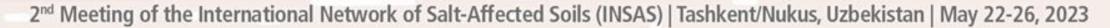
 $[C]^{0} + 2 Cr_{2}O_{7}^{2} + 16 H^{+} = 3 CO_{2} + 4 Cr^{3+} + 8 H_{2}O$





Modifications of the Tyurin method (Russia)

Conditions for the	ovidation	Quantity esti	nation method	
Conditions for the	oxidation	Cr ³⁺	Cr ₂ O ₇ ²⁻	Reference documents
Temperature	Time	Colorimetric	Titrimetric	
100 °C (water bath)	1 hour	λ = 590 nm (Mohr's salt)	-	Turin, 1931; GOST 26213-91
>140 °C (electric stove)	5 min	-	titrant - Mohr's salt solution	Simakov, 1957
>140 °C (electric stove)	5 min	λ = 590 nm (Mohr's salt)	-	Orlov, 1967
20 °C	24 hours	λ = 590 nm (Mohr's salt)	-	Samoilova, Rogiznaya, 2013
>140 °C (water bath)	20 min	λ = 590 nm (sucrose)	-	Simakov, Tsyplakov, 1969
150 °C (drying cabinet)	20 min	λ = 590 nm (sucrose)	titrant - Mohr's salt solution	Nikitin, 1983







Modifications of the Tyurin method (Russia)

Conditions for the	ovidation	Quantity estim		
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Tomnoraturo	Time	Colorimetric	Titrimetric	



Harmonization of soil research methods is the main strategy of the Global Soil Laboratories Network GLOSOLAN

https://www.fao.org/global-soil-partnership/glosolan/

>140 °C (water bath)	20 min	λ = 590 nm (sucrose)	-	Simakov, Tsyplakov, 1969
150 °C (drying cabinet)	20 min	λ = 590 nm (sucrose)	titrant - Mohr's salt solution	Nikitin, 1983



Organic carbon

Carbon, as soil organic matter, alters the physical (e.g. structure), chemical (e.g. cation exchange capacity), and biological (e.g. microbial activity) properties of soils with impacts on plant growth and yield, biodiversity and the soil water retention capacity. The content of organic carbon of mineral horizons can be used also in soil classification, taking the textural class into account. However, the inferred organic carbon status of a soil should always be locally checked as it is only a rough estimate.

The methods to measure organic carbon are rather easy to run but a special effort should be made by soil analysis laboratories to provide the best possible quality data. This will allow monitoring of changes in SOC at both local and regional scales and also give a better idea of the future scenarios, not only for SOC content but also for atmospheric CO₂ evolution. Did you know that the Global Soil Partnership launched a series of activities on soil organic carbon? For more information click here.

The methods to quantify SOC already harmonized by GLOSOLAN are the following:

SOP Walkley-Black method – titration and colorimetric method (EN | ES | RU)

Soil organic carbon – Tyurin colorimetric method (EN | RU)

Training video: Walkley and Black - titration method

Training video: Walkley and Black - colorimetric method

Soil Organic Carbon methods : Sustainability of methods

Method	Risk for human health related to the use of chemicals and the overall implementation of procedure by staff	Environmental risk (waste disposal)	Level of technology required	Average duration of the analysis	Global median price of the analysis (for the customers)		
Walkley & Black	High	High	Low	Up to one working day	6 USD		
Tyurin	High	High	Low	Up to one working day	7.6 USD		

https://www.fao.org/global-soil-partnership/glosolan/ru/







The task of the PT participant



from 1 to 4 methods depending on the capabilities of laboratories

Nº	Methods	Units of measure	S	oil	Α	S	oil	В	S	oil	С	S	oil	D	S	oil	Ε	S	oil	F	Sc	oil	G	Sc	bil	Η	S	oil	Ι
1	LOI	%	A1 ₁	A1 ₂	A1 ₃	B1 ₁	B1 ₂	B1 ₃	C1 ₁	C1 ₂	C1 ₃	D1 ₁	D1 ₂	D1 ₃	E1 ₁	E1 ₂	E1 ₃	F1 ₁	F1 ₂	F1 ₃	G1 ₁	G1 ₂	G1 ₃	H1 ₁	H1 ₂	H1 ₃	11 ₁	11 ₂	11 ₃
2	DC	%	A2 ₁	A2 ₂	A2 ₃	B2 ₁	B2 ₂	B2 ₃	C2 ₁	C2 ₂	C2 ₃	D2 ₁	D2 ₂	D2 ₃	E2 ₁	E2 ₂	E2 ₃	F2 ₁	F2 ₂	F2 ₃	G2 ₁	G2 ₂	G2 ₃	H2 ₁	H2 ₂	H2 ₃	12 ₁	12 ₂	12 ₃
3	WB	%	A3 ₁	A3 ₂	A3 ₃	B3 ₁	B3 ₂	B3 ₃	C3 ₁	C3 ₂	C3 ₃	D3 ₁	D3 ₂	D3 ₃	E3 ₁	E3 ₂	E3 ₃	F3 ₁	F3 ₂	F3 ₃	G3 ₁	G3 ₂	G3 ₃	H3 ₁	H3 ₂	H3 ₃	13 ₁	13 ₂	13 ₃
4	Т	%	A4 ₁	A4 ₂	A4 ₃	B4 ₁	B4 ₂	Β4 ₃	C4 ₁	C4 ₂	C4 ₃	D4 ₁	D4 ₂	D4 ₃	E4 ₁	E4 ₂	E4 ₃	F4 ₁	F4 ₂	F4 ₃	G4 ₁	G4 ₂	G4 ₃	H4 ₁	H4 ₂	H4 ₃	141	142	14 ₃



The task of the PT participant



from 1 to 4 methods depending on the capabilities of laboratories

Each laboratory will receive from us:	Nº	Methods	Units of measure	S	oil	Α	S	oil	B	S	oil	С	S	oil	D	S	oil	Ε	S	oil	F	So	oil	G	Sc	oil	Η	S	oil	Ι
3 WB % $A3_1 A3_2 A3_3 B3_1$ Each laboratory will receive from us:	1	LOI	%	A1 ₁	A1 ₂	A1 ₃	B1 ₁	B1 ₂	B1 ₃	C1 ₁	C1 ₂	C1 ₃	D1 ₁	D1 ₂	D1 ₃	E1 ₁	E1 ₂	E1 ₃	F1 ₁	F1 ₂	F1 ₃	G1 ₁	G1 ₂	G1 ₃	H1 ₁	H1 ₂	H1 ₃	11 ₁	11 ₂	11 ₃
$\frac{3_{3}}{3_{1}} = \frac{3_{3}}{3_{1}} = \frac{3_{3}}{3$	2	DC	%	A2 ₁	A2 ₂	A2 ₃	B2 ₁	B2 ₂	B2 ₃	C2 ₁	C2 ₂	C2 ₃	D2 ₁	D2 ₂	D2 ₃	E2 ₁	E2 ₂	E2 ₃	F2 ₁	F2 ₂	F2 ₃	G2 ₁	G2 ₂	G2 ₃	H2 ₁	H2 ₂	H2 ₃	12 ₁	12 ₂	12 ₃
4 T % A4 A4 B4 Each laboratory will receive from us:	3	WB	%	A3 ₁	A3 ₂	A3 ₃	B3 ₁																				33	13 ₁	13 ₂	13 ₃
	4	Т	%	A4 ₁	A4 ₂	A4 ₃	84 ₁	E	ac	h la	abc	ora	tor	y v	vill	re	cei	ve	trc	m	US	•					4 ₃	14 ₁	14 ₂	I4 ₃

- 9 containers with soil (A, B, C, D, E, F, G, H, I),
- Invitation,
- Instructions



Before the start of PT



Food and Agriculture Organization of the General instructions How to produce a soil sample for the proficiency testing of the Global Soil Laboratory Network - GLOSOLAN -

Preparation of soil samples for PT





Before the start of PT



Homogeneity

We carry out BEFORE mailing the soil to the participating laboratories. 10 times 2 parallel repetitions for each sample for each method. = 20 measurements for each sample for each method.





During PT



Stability

which determines the period life of the soils.

10 times 1-2 parallel replicates for each sample for each method.

= 10-20 measurements for each sample for each method.

The measurement period is from the beginning of PT to the end of PT. 1 or 2 times a week.

Later we will calculate the frequency of measurements





After PT



Statistical (Math) analysis of results

Using the GLOSOAN platform.

Online tools



GLOSOLAN PT results submission platform





PT results

1. RELIABILITY: for each lab, what is its precision?

when analysing several times the same sample, how close are the results?

2. COMPARABILITY: among all labs, dispersion of their results?

when the same sample is analysed by several labs, how close are the results?



What is the quality of the analysis? How to improve the quality of analysis?



Invitation



FIRST INFORMATION LETTER on GLOSOLAN Proficiency test for Eurasia 2023

Dear Colleagues,

We invite your laboratory to take part in GLOSOLAN Proficiency test (PT) for Eurasia 2023 on the measurement of soil organic carbon (SOC) organized by the Institute of Biology of the Komi Science Center of the Ural Branch of the Russian Academy of Sciences (IB) with the financial support of the partner PhosAgro.

Our experience in measuring SOC and the study of the most common and technically available methods allowed us to certify the methods, No. 88-17641-001-2020, FR.1.31.2020.32218). A comparative study of standard and different soil types samples carried out by the IB staff has allowed establishing the interelationship between SOC coltained by the use of the photometric method and the dry combustion method on the analyzer. The modification of the Tyruin method was approved by the GLOSOLAN commission, the description of the methods is available on the official website: - Walkley-Black method in English: <u>https://www.fao.org/3/ca7471eu/ca7471ru/ca7471ru.pdf</u> - Walkley-Black method in English: <u>https://www.fao.org/3/ca7471ru/ca7471ru.pdf</u> - Tyruin method in English: <u>https://www.fao.org/3/ca7471ru/ca7471ru.pdf</u> - Tyruin method in Russian: <u>https://www.fao.org/3/ca7471ru/ca7471ru.pdf</u> - Tyruin method in Russian: <u>https://www.fao.org/3/ca7471ru/ca7471ru.pdf</u> - Tyruin method in Russian: <u>https://www.fao.org/3/ca7471ru/ca7471ru.pdf</u> - video on the Tyruin method in Black in English: <u>https://www.youtube.com/watch?v=W2b60504TM |</u> <u>https://wwww.youtube.com/watch?v=W2b60504TM |</u> <u>https://www.yout</u>

The purpose of the organized P1s is to expand and promote common approaches for measuring SOC into the laboratories' practice.

In connection with the above, we prepared a selection of seven control non-calcareous soil samples based on real objects for measuring SOC. Samples will be sent to participants with following accompanying documentation: -user's manual:

-certified method for measuring the carbon mass fraction of organic compounds and organic matter in soils, ground material, parent material, bottom sediments by the photometric method (Tyurin and Walkley-Black methods) No. 88-17641-001-2020 (FR.1.31.2020.38218); - the form of issuing measurement results.

In control samples, it will be necessary to determine the SOC by all the proposed methods (recommended) or to choose any of them if a limited technical capacity doesn't allow conducting studies using all methods.

GLOSOLAN Proficiency test for Eurasia 2023 are held **free of charge**. All PT participants will be provided with a final report in which test results from participating laboratories will be **prevented anonymously**. Every participant will additionally receive an individualPT participation certificate. PARTICIPANT REGISTRATION FORM GLOSOLAN proficiency test for Eurasia 2023

ame of the lab

Name of the laboratory		
Unique accreditation record		
number in the accredited persons		
registry (if available)		
Organization name		
Country		
Registered address		
Place (address) of activity		
Adress for sending control		
samples		
Full name, position, phone		
number, e-mail of the person		
responsible for interaction within		
the PT		
The prior measuring method by	Declared research method	Choose
The prior measuring method by which the laboratory will	Tyurin method for measuring SOC by the photometric	Choose Yes/No
The prior measuring method by which the laboratory will conducting studies	Tyurin method for measuring SOC by the photometric method according to the measurement procedure No.	
The prior measuring method by which the laboratory will conducting studies (choose any number of research	Tyurin method for measuring SOC by the photometric	
The prior measuring method by which the laboratory will conducting studies (choose any number of research methods, depending on technical	Tyurin method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-2020 (FR.1.31.2020.38218) Walkley-Black method for measuring SOC by the	
The prior measuring method by which the laboratory will conducting studies (choose any number of research	Tyurin method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-2020 (FR.1.31.2020.38218)	Yes/No
The prior measuring method by which the laboratory will conducting studies (choose any number of research methods, depending on technical	Tyurin method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-2020 (FR.1.31.2020.38218) Walkley-Black method for measuring SOC by the	Yes/No
The prior measuring method by which the laboratory will conducting studies (choose any number of research methods, depending on technical	Tyurin method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-2020 (FR.1.31.2020.38218) Walkley-Black method for measuring SOC by the photometric method according to the measurement	Yes/No
The prior measuring method by which the laboratory will conducting studies (choose any number of research methods, depending on technical	Tyurin method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-2020 (FR.1.31.2020.38218) Walkley-Black method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-2020	Yes/No
The prior measuring method by which the laboratory will conducting studies (choose any number of research methods, depending on technical	Tyurin method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-2020 (FR.1.31.2020.38218) Walkley-Black method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-2020 (FR.1.31.2020.38218)	Yes/No Yes/No
The prior measuring method by which the laboratory will conducting studies (choose any number of research methods, depending on technical	Tyurin method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-2020 (FR.1.31.2020.38218) Walkley-Black method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-2020 (FR.1.31.2020.38218) Dry combustion according to the methodology carried	Yes/No Yes/No
The prior measuring method by which the laboratory will conducting studies (choose any number of research methods, depending on technical capacity, delete as appropriate) Your comments, suggestions and	Tyurin method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-0202 (FR.1.31.2020.38218) Walkley-Black method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-2020 (FR.1.31.2020.38218) Dry combustion according to the methodology carried out in the daily laboratory activities of the participant	Yes/No Yes/No Yes/No
The prior measuring method by which the laboratory will conducting studies (choose any number of research methods, depending on technical capacity, delete as appropriate)	Tyurin method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-0202 (FR.1.31.2020.38218) Walkley-Black method for measuring SOC by the photometric method according to the measurement procedure No. 88-17641-001-2020 (FR.1.31.2020.38218) Dry combustion according to the methodology carried out in the daily laboratory activities of the participant	Yes/No Yes/No Yes/No

The completed form need to send on the organizer e-mail soiltest@ib.komisc.ru no later than June 15, 2023.





Each laboratory will be assigned a code.

Participation in PT is anonymous.

PT is held free of charge.



Invitation



IMPORTANT DATES

EVENT	DATE
First Information letter	05.05.2023
Registration of PT participants. Acceptance of participants forms	10.05-15.06.2023 г.
	deadline 15.06.2023
Second Information Letter	16.06.2023
Online webinar on the organization of PT, including training on each method	16.06.2023
Sending of control soil samples to participants	16-30.06.2023 г.
Measuring procedures	01.07-30.09. 2023
Provision of measuring results	deadline - 30.09.2023
Statistical (Math) analysis of the results and final report preparation, provision	30.09-31.10.2023
of personal reports and PT participant certificates	

Feel free to contact us for more information - soiltest@ib.komisc.ru.

CLOBAL SOIL

Prospective PT participants



- Kazakhstan,
- Russia (14 RUSOLAN's laboratories),
- Turkmenistan,
- Uzbekistan,

and possibly

- Armenia,
- Azerbaijan,
- Georgia, and Kyrgyzstan

There is interest in **Tajikistan**, but there is no suitable laboratory



Prospective PT participants



- Kazakhstan,
- Russia (14 I
- Turkmenist
- Uzbekistan

We invite you to participate in the PT!



There is interest in Tajikistan, but there is no suitable laboratory



Prospective PT participants



- Kazakhstan,
- Russia (14 I
- Turkmenist
- Uzbekistan

We invite you to participate in the PT!



Dissemination of information about the PT

- GLOSOLAN,
- RUSOLAN (ib.komisc.ru/rusolan)

is no suitable laboratory





Subregional Eurasian $PT \rightarrow Search$ for transfer functions

Data used:

- Homogeneity,
- Stability,
- GLOSOLAN Proficiency test for Eurasia 2023,
- published data obtained at the Russian National Reference Laboratory (Syktyvkar),
- Global PT?







Subregional Eurasian PT \rightarrow Search for transfer functions

Data used:

- Homogen
- Stability,
- We invite you to become part of the strategic process!
- GLOSOLA
- published uata obtained at the Nussian National Neterence Laboratory (Syktyvkar),
- Global PT?







Food and Agriculture Organization of the United Nations

Thank you for your attention!

<u>shamrikovaelena@yandex.ru</u> ib.komisc.ru/rusolan

