



Food and Agriculture
Organization of the
United Nations

5th Meeting of the **European and Eurasian Soil Laboratory Network** (EUROSOLAN)

18-19 October 2023



Eurasian proficiency test (PT) 2023

Presenter:

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EUROSOLAN vice-Chair for Eurasia

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EUROSOLAN

EUROPEAN AND EURASIAN SOIL LABORATORY NETWORK



Main authors at the Institute of Biology of Komi Scientific Center of the Ural Branch of the RAS, Syktyvkar (in alphabetical order):



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

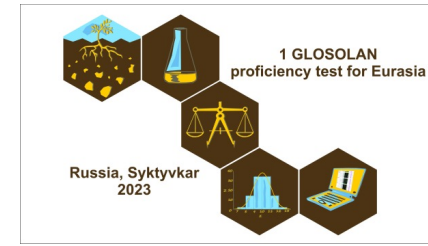
Svetlana Kostrova – Lead chemical engineer

Tatyana Zonova – Lead chemical engineer

Yulia Bobrova – Lead chemical engineer



Why does a testing laboratory need to participate in Proficiency tests?



Main outcome of any testing laboratory (*it don't have to be accredited*) is a measurement result.

Laboratory's task is not only **to guarantee a quality** of each result, but also to show **evidence**.

By participating in PT, each laboratory **can provide evidence** of its competence to

- its colleagues,
- clients,
- interested parties,
- and accreditation body.

Eurasian PT-2023 concept



Coordinator PT – NatRef Lab of the RUSOLAN

Participation in PT is **free and anonymous**.

The purpose of the PT: to expand and promote unified approaches for measuring SOC into the laboratories' practice.

We noticed that the laboratories :

- afraid to participate in PT (even anonymously),
- and don't see the point in participating - *why?*

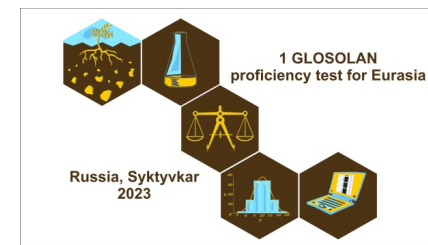
We need to work with this!

Eurasian PT-2023 concept

PT participants*

- Armenia (1)
- Georgia (1)
- Kazakhstan (2)
- Moldova (1)
- Belarus (1)
- Russia (15 RUSOLAN's labs)
- Senegal (1)
- Turkmenistan (1)
- Uzbekistan (3)

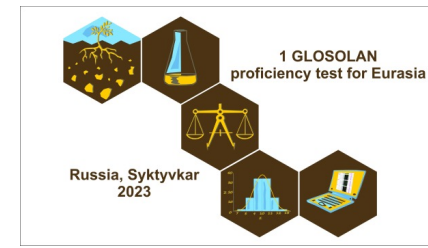
* - *with the support of Maria Konyushkova*



The vast majority of laboratories:

- not accredited,
- never participated in PT,
- never used the WB method

Eurasian PT-2023 concept



The purpose of the PT: to expand and promote unified approaches for measuring SOC into the laboratories' practice.

PT tasks:

- quality control of measurements,
- identifying problems in laboratories (if any),
- *informational and educational function,*
- *building laboratory capacity (use of new methods)*

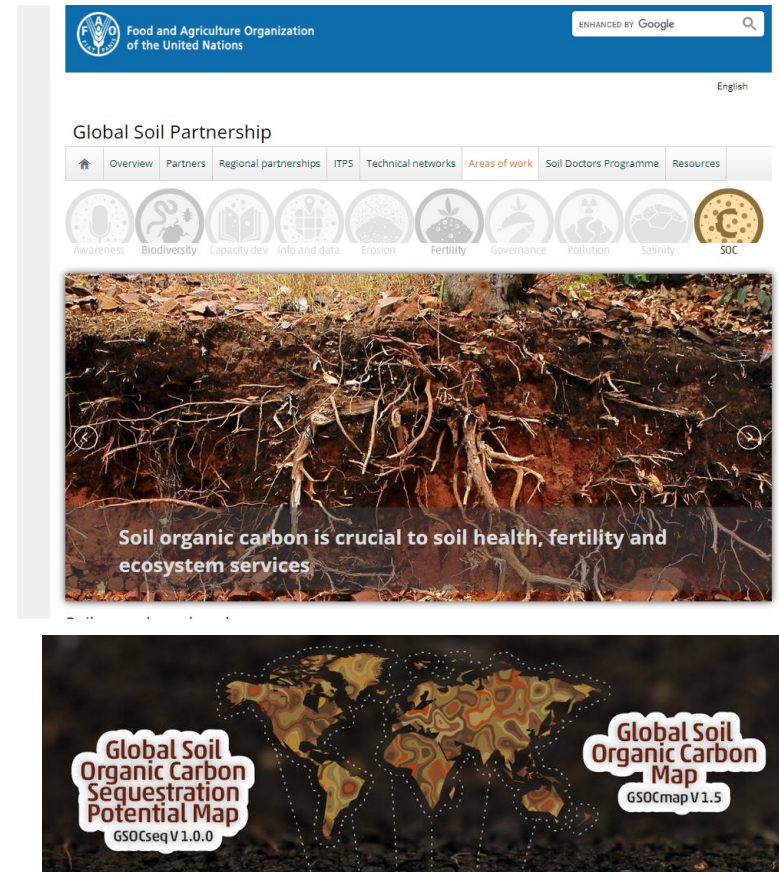
Why SOC?

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.

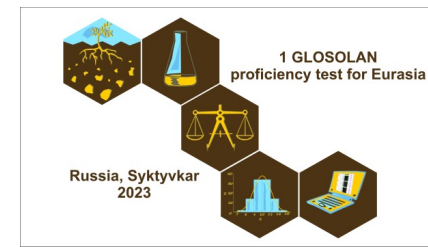
Under the GSP initiative of the FAO, the availability of **non-harmonized data** is one of the reasons for the low accuracy of the global SOC map (*Peralta et al., 2022*).

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

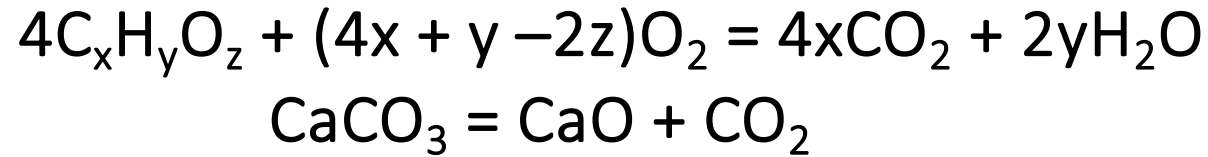


Tested methods

- Dry combustion on the analyzer (**DC**),
- Walkley-Black's method (**WB**),
- Tyurin's method (**T**),
- Loss-on-ignition method (**LOI**)



Dry combustion on the analyzer



Advantages:

- measuring range %C_{tot} from 0.1 to 100%,
- high accuracy of measurement results:
 $\pm\delta = 23, 15, 10$ и $3,5\%$ для %C_{tot} = (01–2); (2–5) и (5–30) и $>30\%$,
- complete oxidation of carbon of organic and inorganic compounds,
- availability of standard samples for analyzer calibration,
- rapidity (batch up to 100 samples),
- selectivity

Reference
method

Disadvantages:

High cost of the device, consumables and maintenance

Dichromatometric method (T, WB)

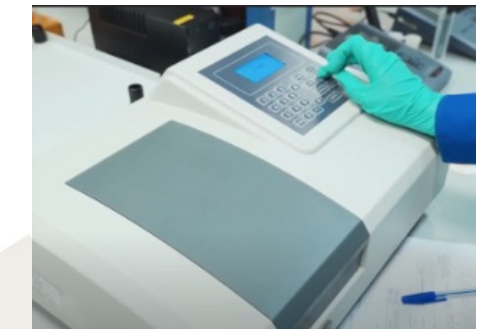
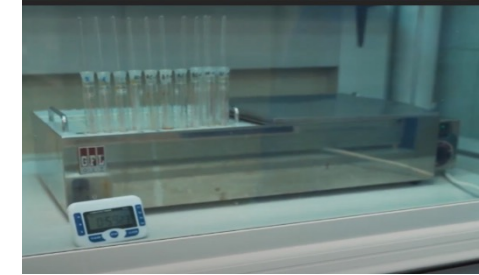
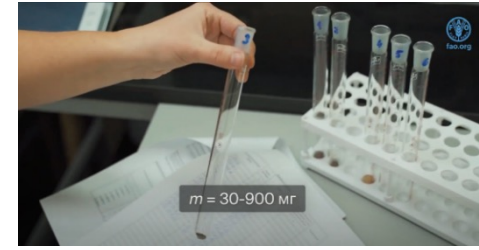
Advantages:

- cheapness,
- significant amounts of data on the world's soils

Disadvantages:

- limited measurement range %Corg from **0.17 to 8.7%**,
- incomplete oxidation of carbon of organic compounds (taking into account the incomplete oxidation of Corg - using universal f),
- labor intensity,
- toxicity.

Relative measurement error: $\pm\delta = 20\%$.



Loss-on-ignition method

Advantages:

- cheapness,
- measuring range %C_{tot} from 0.1 to 100%,
- rapidity

Disadvantages:

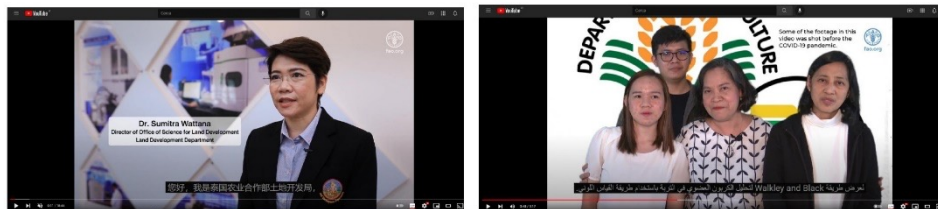
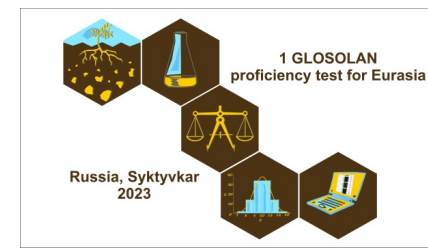
- ?? f for SOM \rightarrow SOC (SOC = SOM / 1.724),
- the presence of mineral compounds that decompose at $T = 105-550^{\circ}\text{C}$ with the release of gaseous products



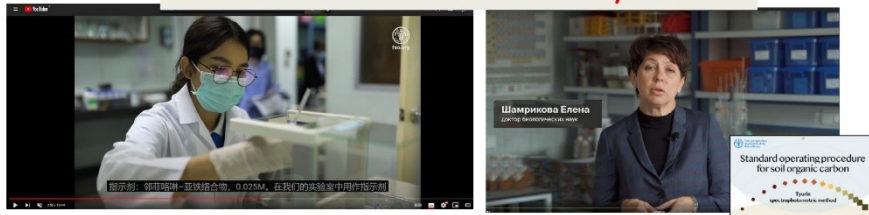
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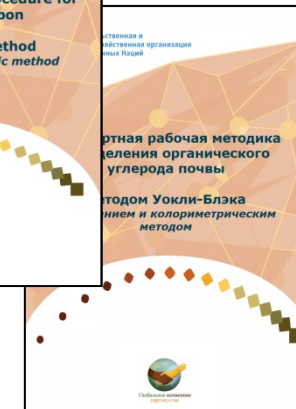
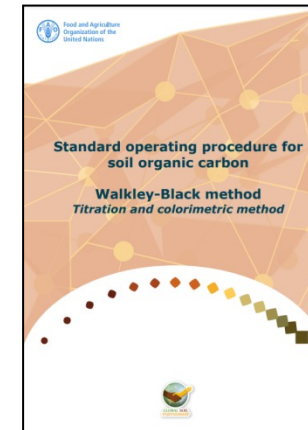
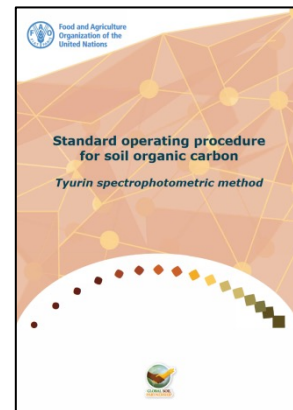
SOPs of GLOSOLAN



Almost 9000 views in one year!



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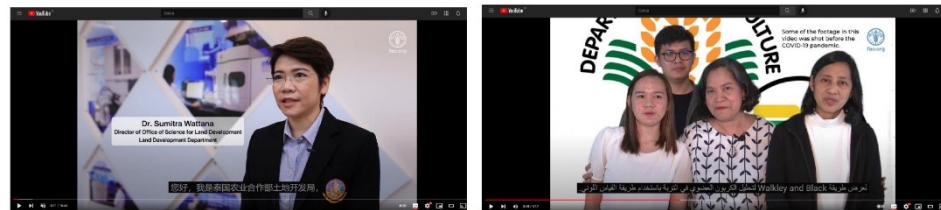
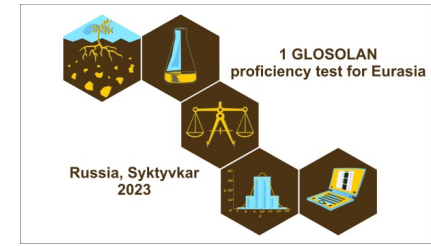
5th Meeting of the **European and Eurasian Soil Laboratory Network (EUROSOLAN)** | 18-19 October 2023



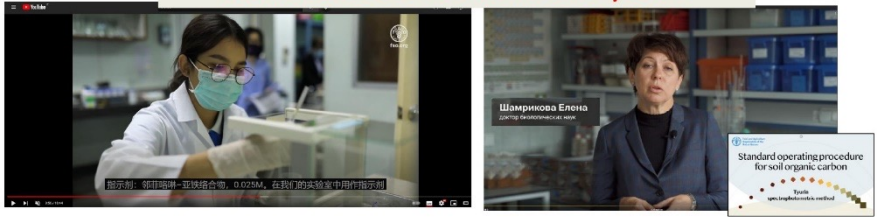
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$$DC = WB \cdot 1.3 = T \cdot 1.15 (P = 0.95)$$

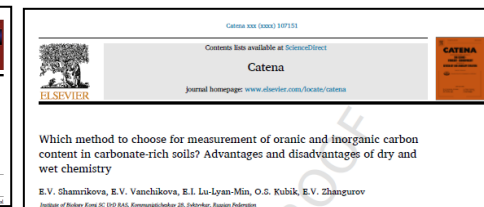
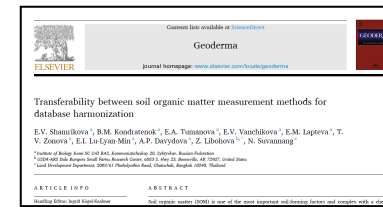
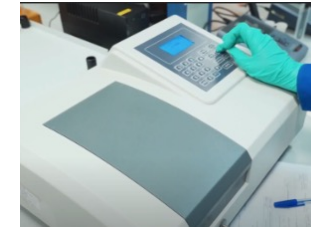


! In the Walkley-Black method, the amount of $K_2Cr_2O_7$ and H_2SO_4 is equal to the same characteristics as in the Tyurin method, but the concentration of these components of the mixture is 1.5 times higher

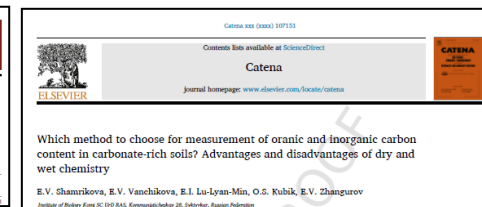
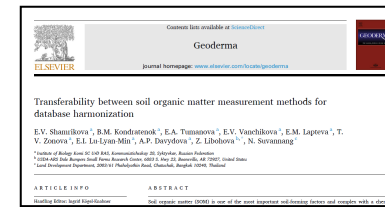
!! Heating of the reaction mixture occurs due to the exothermic effect that occurs when a concentrated solution of H_2SO_4 is mixed with distilled water.



In the Tyurin method, compared to WB, additional dispersion of the solid phase occurs



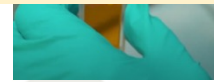
$$DC = WB \cdot 1.3 = T \cdot 1.15 (P = 0.95)$$



In the Walkley-Black method, the amount of $K_2Cr_2O_7$ and H_2SO_4 is equal to the same characteristics as in

PT tasks:

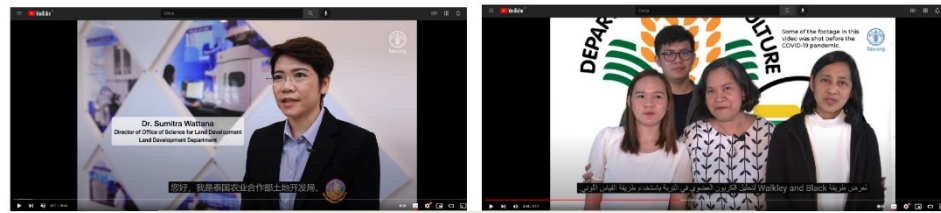
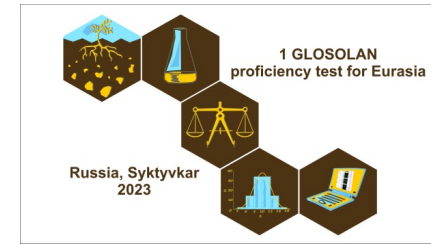
- quality control of measurements using various methods,
- identifying problems in laboratories (if any),
- *informational and educational function,*
- *expanding the competencies of laboratories (use of new methods),*
- *checking procedures and errors in the modification of the Tyurin method developed by our team.*



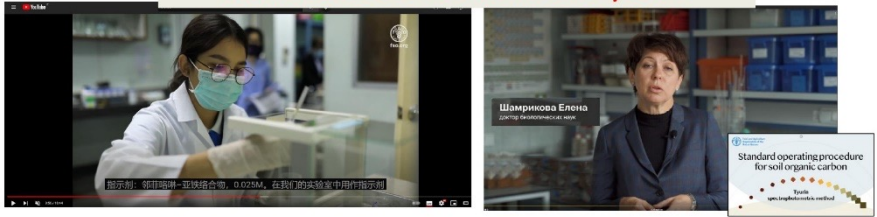
Tested methods

- Dry combustion on the analyzer (DC) ,
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- Tyurin's method (T) ,
- Loss-on-ignition method (LOI) - *SOP of GLOSOLAN in progress*

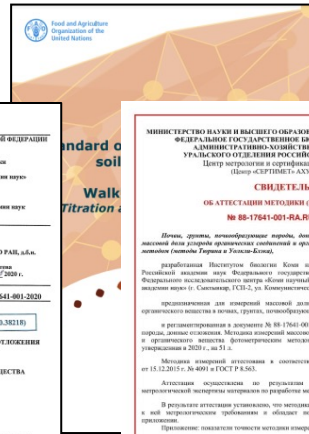
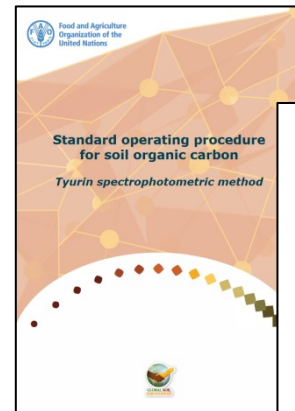
SOPs of GLOSOLAN



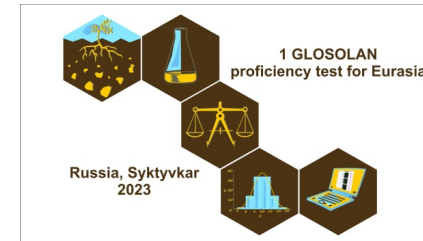
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LOI: SOP of GLOSOLAN in progress



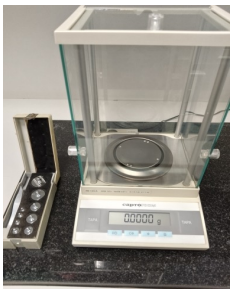
The bad news - LOI has many modifications!

$$T, t, m_{soil}, d$$

Minimize laboratory errors - the task of the PT coordinator.

Uniform LOI conditions.

The conditions were founded **experimentally.**

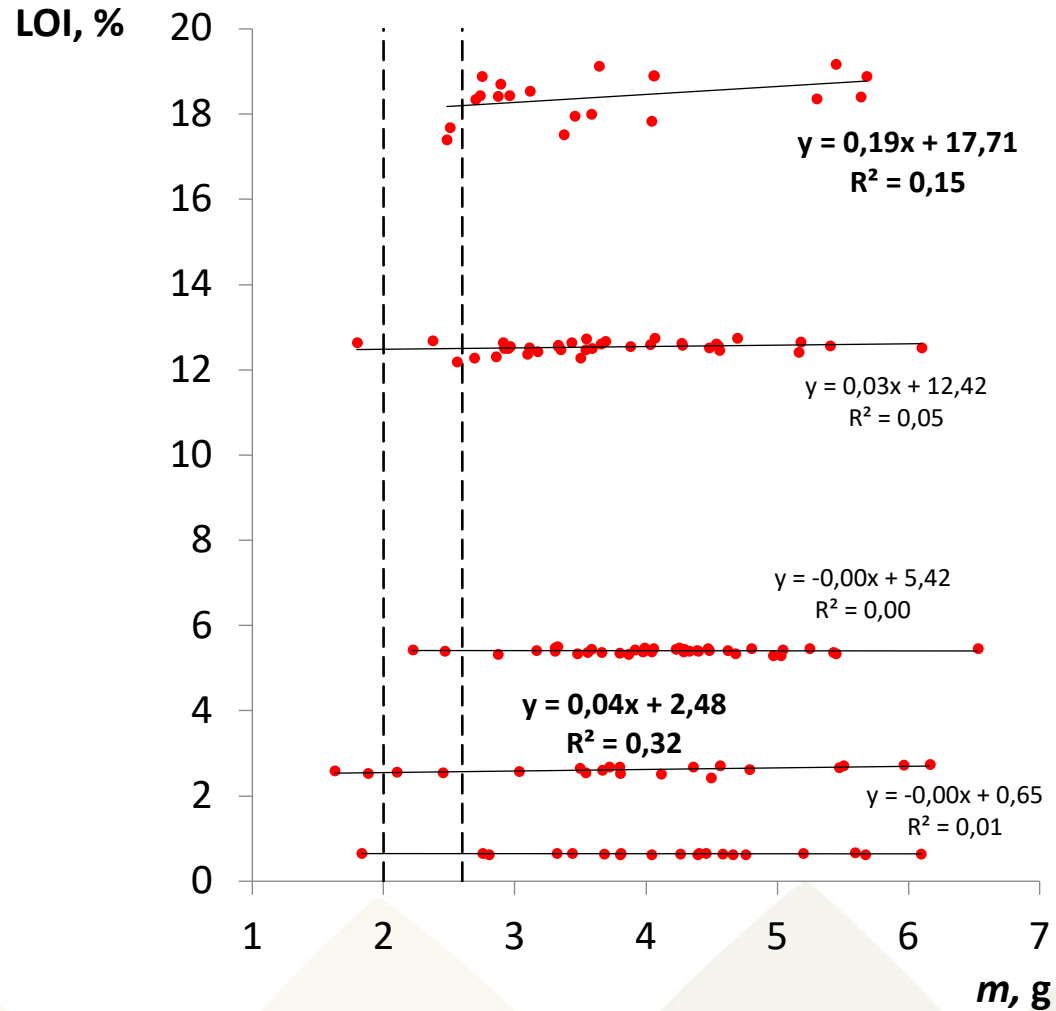
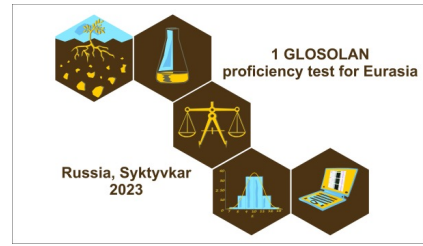


LOI: Heating time

Soil	Heating time, hour											
	T = 105 °C						T = 550 °C					
	4	5	6	7	8	9	6	7	8	9	10	12
1	14,8309	14,8300	14,8296	14,8297	14,8296	14,8297	14,0339	14,0338	14,0339	14,0338	14,0339	14,0339
2	14,4856	14,4831	14,4822	14,4823	14,4823	14,4823	14,2672	14,2673	14,2672	14,2672	14,2672	14,2673
3	17,4958	17,4921	17,4914	17,4914	17,4913	17,4914	16,6163	16,6164	16,6163	16,1663	16,1663	16,1662
4	16,2597	16,2571	16,2561	16,2562	16,2562	16,2562	12,9443	12,9440	12,9440	12,9440	12,9440	12,9439
5	16,0904	16,0867	16,0807	16,0803	16,0803	16,0802	17,9249	17,9236	17,9237	17,9237	17,9236	17,9231
6	19,5486	19,5407	19,5351	19,5346	19,5346	19,5346	16,3069	16,3054	16,3054	16,3054	16,3054	16,6042
7	18,0275	18,0266	18,0263	18,0261	18,0262	18,0262	17,2339	17,2335	17,2336	17,2335	17,2333	17,2330
8	16,8399	16,8393	16,8391	16,8389	16,8389	16,8389	13,1658	13,1656	13,1656	13,1656	13,1654	13,1650

T = 550 °C, t = 7 hours

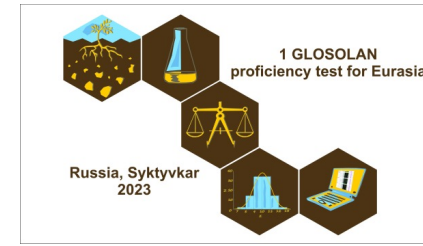
LOI: The mass of the air-dry soil



1. $T = 550\text{ }^{\circ}\text{C}$,
2. $t = 7\text{ hours}$,
3. $m_{soil} = 2,0\text{-}2,5\text{ g}$,



LOI: Sample preparation



7. Sample preparation DC – SOP of GLOSOLAN

Follow the sample preparation instructions provided by the manufacturer for use of the autoanalyzer. Probably, a representative portion of the soil sample that was previously treated (dried and sieved to 2 mm) must be porfirised (grind fine and homogeneously) until the entire fraction passes through a sieve of inferior size. Typically, a representative subsample is taken from the bulk sample and milled to a sufficiently fine mesh size. Ensure that milling equipment and sieves do not introduce contamination to the samples.

WB – SOP of GLOSOLAN

7. Sample preparation

Air dry soil sample and sieve to ≤ 2.0 mm size.

7. Sample preparation T – SOP of GLOSOLAN

Soil samples are prepared under conditions that ensure the composition of the sample in accordance with the regulatory and technical documentation for the objects studied. Air-dry samples of soils are obtained by drying at the temperature and humidity of the laboratory room. A portion of the soil sample is taken and scattered on tracing paper, large inclusions (undecomposed roots and plant debris, stones, tumors, etc.) are removed with tweezers.

The soil sample is sieved (< 1 mm or other diameter, according to the procedures of e... portion is grinded in a jasper or agate mortar to a size of 0.25 mm in diameter. The prep... stored in plastic bags, plastic or glass containers. The mass of the air-dry soil sample to

LOI ???????????

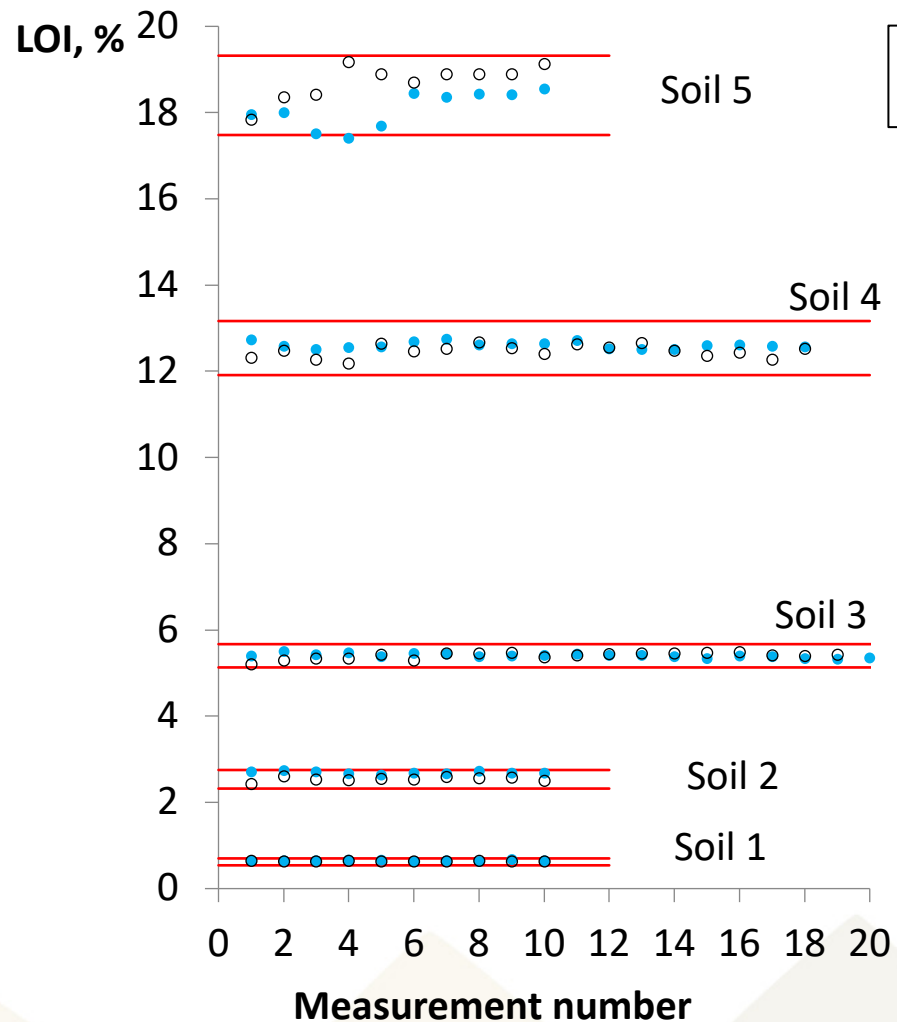
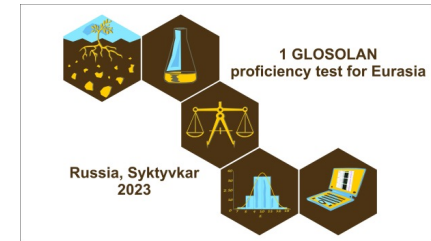
SOP of GLOSOLAN in progress

In Russia, $d = 1$ mm

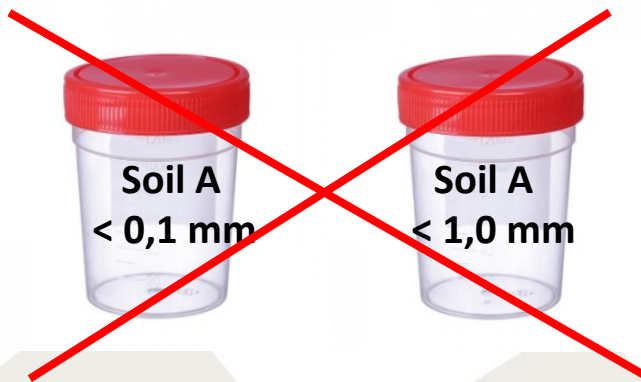
DC, WB, T: $< 0,1$ mm



Sample preparation



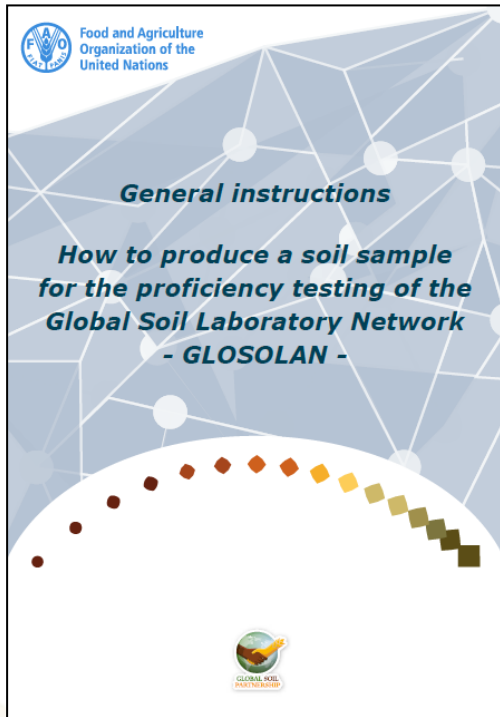
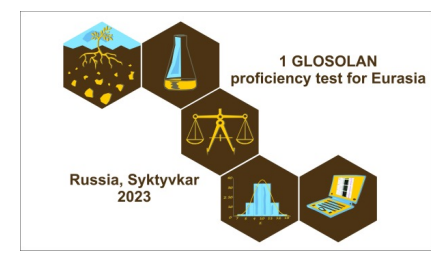
We compared LOI: soils <0,1 mm and <1,0 mm (reproducibility conditions).
Variation results < 5%, great!
DC, WB, T, LOI: < 0,1 mm



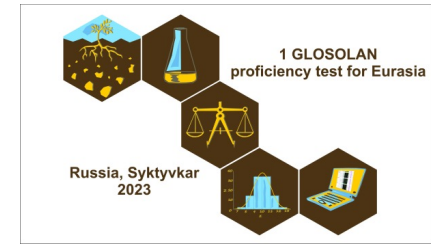
Before the start of PT

Preparation of soil samples for PT

Corg 0,17-8,7%, CaCO3 0%



The task of the PT participant



From 1 to 4 methods depending on the capabilities of laboratories

27 September – 15 November 2023

№	Methods	Units of measure	Soil A			Soil B			Soil C			Soil D			Soil E			Soil F			Soil G			Soil H			Soil I		
			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	I1 ₁	I1 ₂	I1 ₃
1	DC	%	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	I1 ₁	I1 ₂	I1 ₃
2	WB	%	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	I2 ₁	I2 ₂	I2 ₃
3	T	%	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	I3 ₁	I3 ₂	I3 ₃
4	LOI	%	A4 ₁	A4 ₂	A4 ₃	B4 ₁	B4 ₂	B4 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	I4 ₁	I4 ₂	I4 ₃


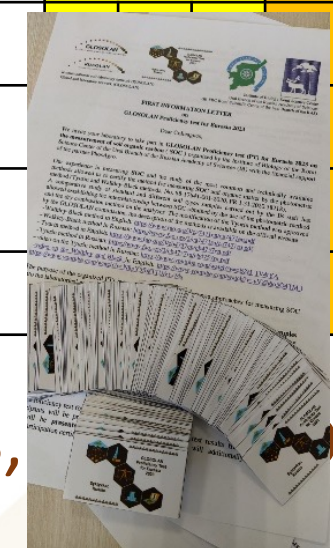



The task of the PT participant

27 September – 15 November 2023

From 1 to 4 methods depending on the capabilities of laboratories

№	of ure	Soil A			Soil B			Soil C			Soil D		Soil E	Soil F	Soil G	Soil H	Soil I
					B1 ₂	B1 ₃		C1 ₁	C1 ₂	C1 ₃	D1 ₁	D1 ₂					
1																	I1 ₃
2					B2 ₂						1	D2					I2 ₃
3					B3 ₂						1	D3					I3 ₃
4					B4 ₂						1	D4					I4 ₃

			<p>Each laboratory received :</p> <ul style="list-style-type: none"> - Information letters (N 1, 2), - SOPs, - Instructions, - Forms for recording results 				
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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?



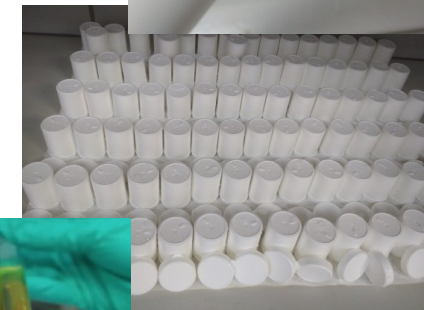
What is the quality of the analysis?
How to improve the quality of analysis?

Homogeneity - Before the start of PT

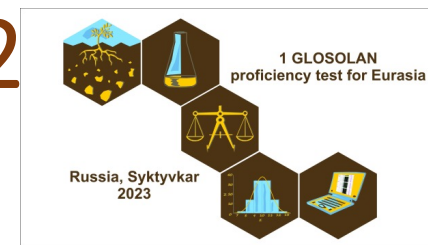
10 times 2 parallel repetitions for each sample for each method
= 20 measurements for each sample for each method

Stability - During PT

10 times 1-2 parallel replicates for each sample for each method
= 10-20 measurements for each sample for each method



Webinar dedicated to holding Eurasian PT-2023 26.09.2023



26 September 2023 10.30 – 12.00 (Moscow time) Link to the event: https://trueconf.ru/c/34d62ba65a7bfc68ceda05d2be8ceae9 Moderator: Elena Shamrikova		
10.30 – 10.35	Greeting: Institute of Biology, Komi Scientific Center, Ural Branch of the RAS	Ph.D. Ivan Chadin, Director
10.35 – 10.50	Experience of the Institute of Biology in Harmonization of Methods for Measuring of Soil Organic Carbon	Dr. Elena Shamrikova, Chairman of RUSOLAN
10.50 – 11.05	Why does a testing laboratory need to participate in Proficiency Tests?	Svetlana Kostrova, Head of the Ecoanalytical Laboratory
11.05 – 11.30	Methodological features of measurements of Soil Organic Carbon in soils in accordance with the Walkley-Black method and a modification of the Tyurin method	Ph.D. Elena Lapteva, Head of the Department of Soil Science, Evgenia Tumanova, Lead Engineer
11.05 – 11.30	Method for measuring Soil Organic Carbon by Loss on Ignition method	N.N. Bondarenko, Engineer of the 1 category, Ph.D. Elena Lapteva, Head of the Department of Soil Science, Elena Kyzurova, Lead Engineer
11.30 – 11.55	Round of questions and answers	Dr. Elena Shamrikova, Chairman of RUSOLAN
11.55 – 12.00	Closing the webinar	

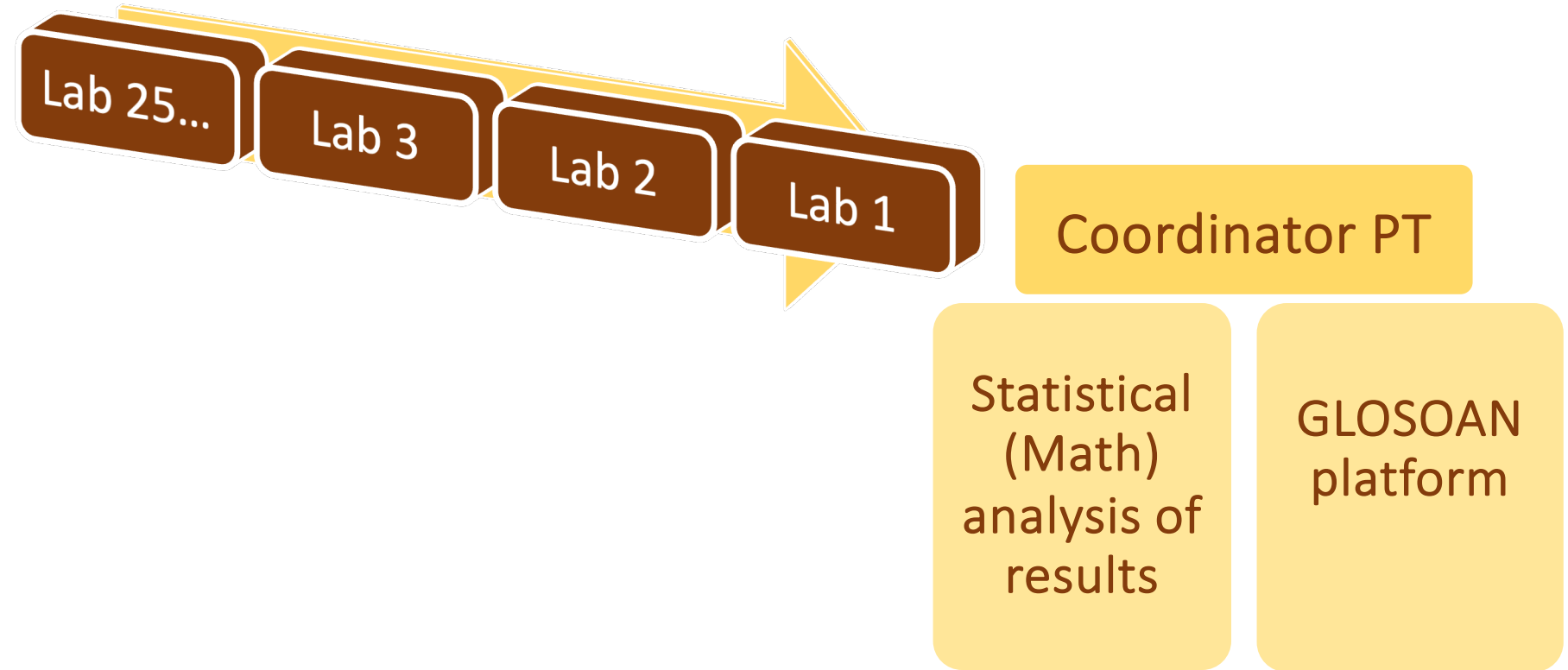


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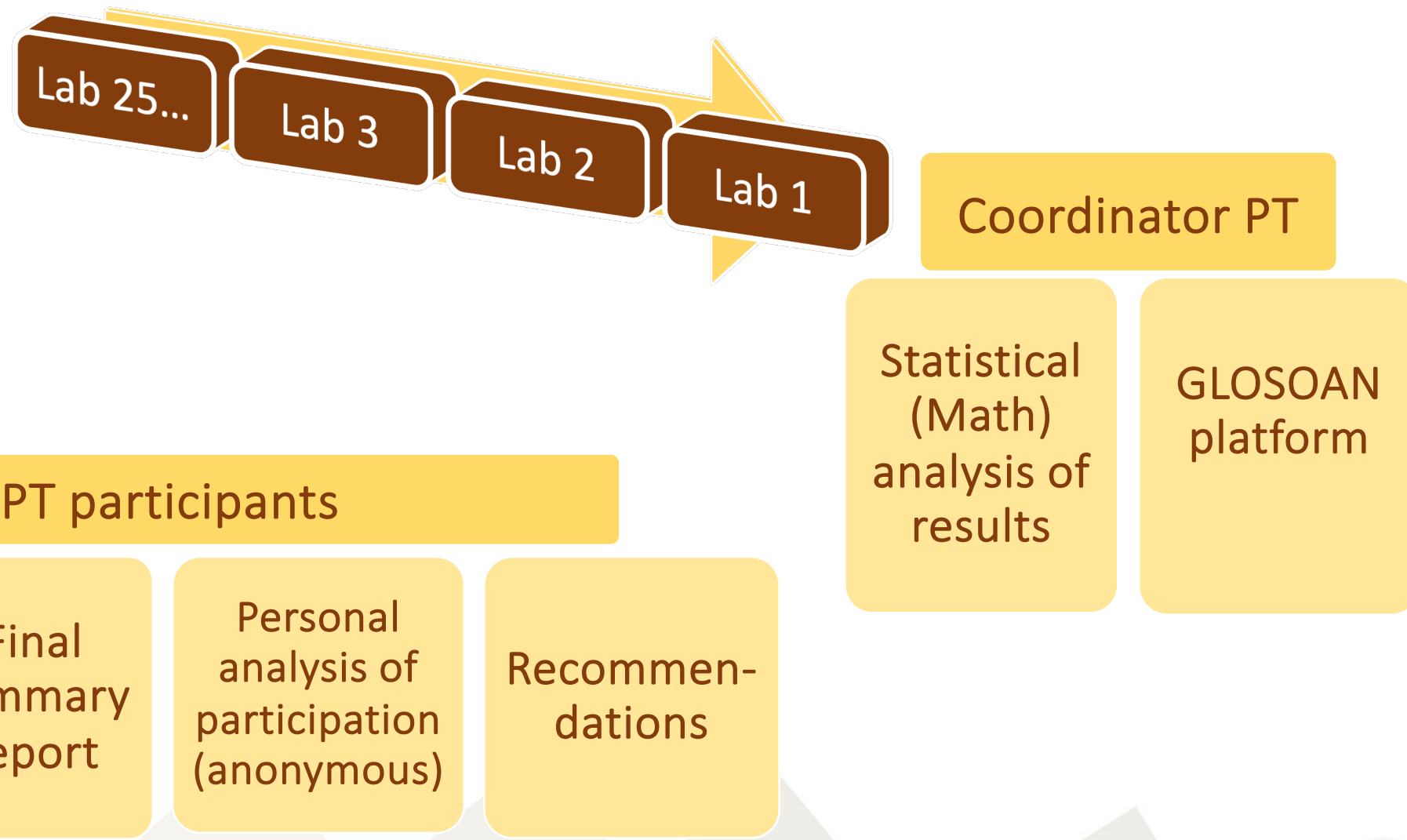
5th Meeting of the **European and Eurasian Soil Laboratory Network (EUROSOLAN)** | 18-19 October 2023



After PT



After PT



Conclusion

PT is the driving force behind the quality control of analyses.

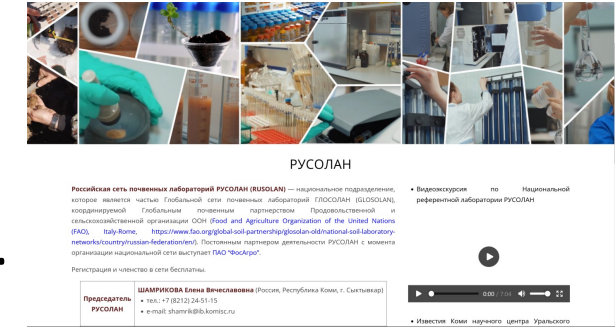
Both coordinators and participants are building capacity:

- quality control,
- mastering new methods,
- harmonization of data,
- expansion of links between laboratories.

At the same time, PT requires significant efforts and resources.

It is necessary to continue educational activities about PT.

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THANK YOU
FOR ATTENTION!

