Results of GLOSOLAN PT for Eurasia 2023. Soil organic carbon. Dry combustion and Loss-on-ignition methods

Webinar

29 October 2024

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Dry combustion on the analyzer

$$4C_xH_yO_z + (4x + y - 2z)O_2 = 4xCO_2 + 2yH_2O$$

$$CaCO_3 = CaO + CO_2$$

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

Disadvantages:

High cost of the device, consumables and maintenance

Loss-on-ignition method

Advantages:

- cheapness,
- measuring range % Ctot from 0.1 to 100%,
- rapidity

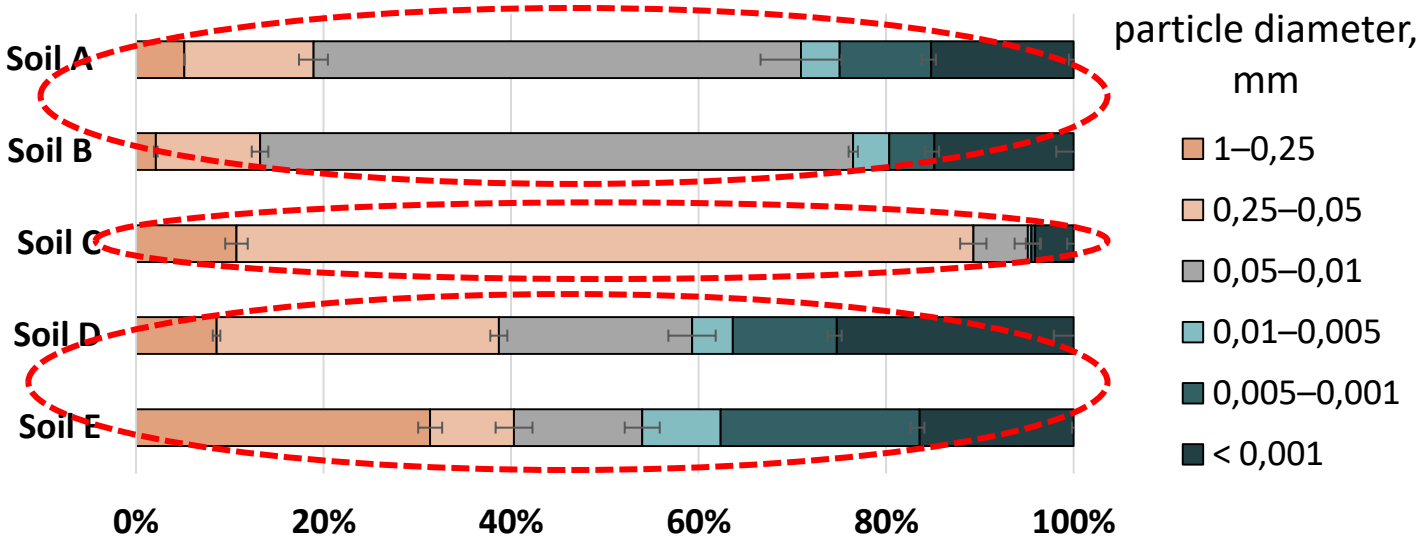
Disadvantages:

- ?? f for SOM → SOC (SOC = SOM /1.724 = SOM *0.58),
- the presence of mineral compounds that decompose at T = 105-550˚C with the release of gaseous products

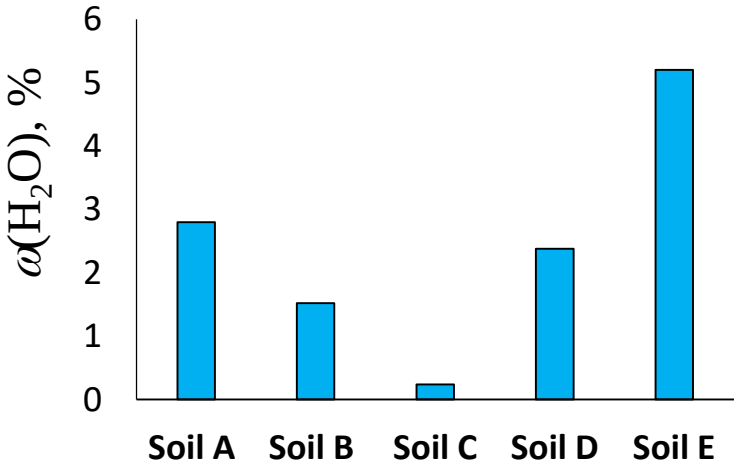
Object of study

Control soils	Depth, cm	Soil type
Soil A = Soil F = Soil I	0-20	Agro-sod-podzolic Loamy
Soil B = Soil G = Soil H	0-20	Agro-sod-podzolic Loamy
Soil C	5(7)-15(20)	Illuvial-ferruginous podzol on two-membered deposits (sand underlain by loams)
Soil D	40-60	Illuvial-ferruginous podzol on two-membered deposits (sand underlain by loams)
Soil E	5-18	Sod-gley clay

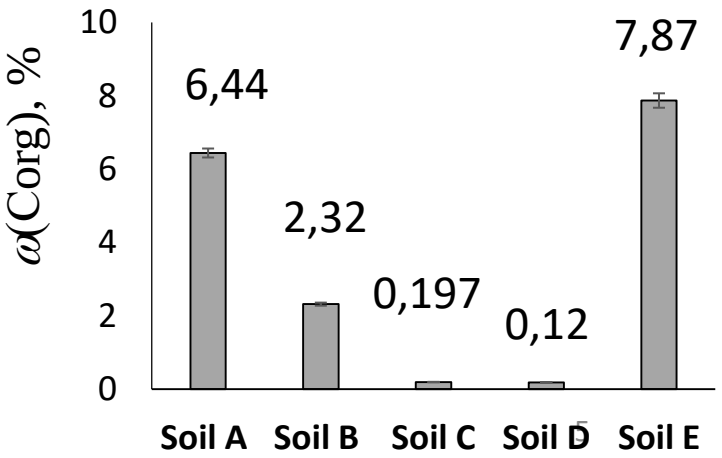
Particle size distribution of soils



Mass fraction of hygroscopic water, %



Mass fraction of Corg, %

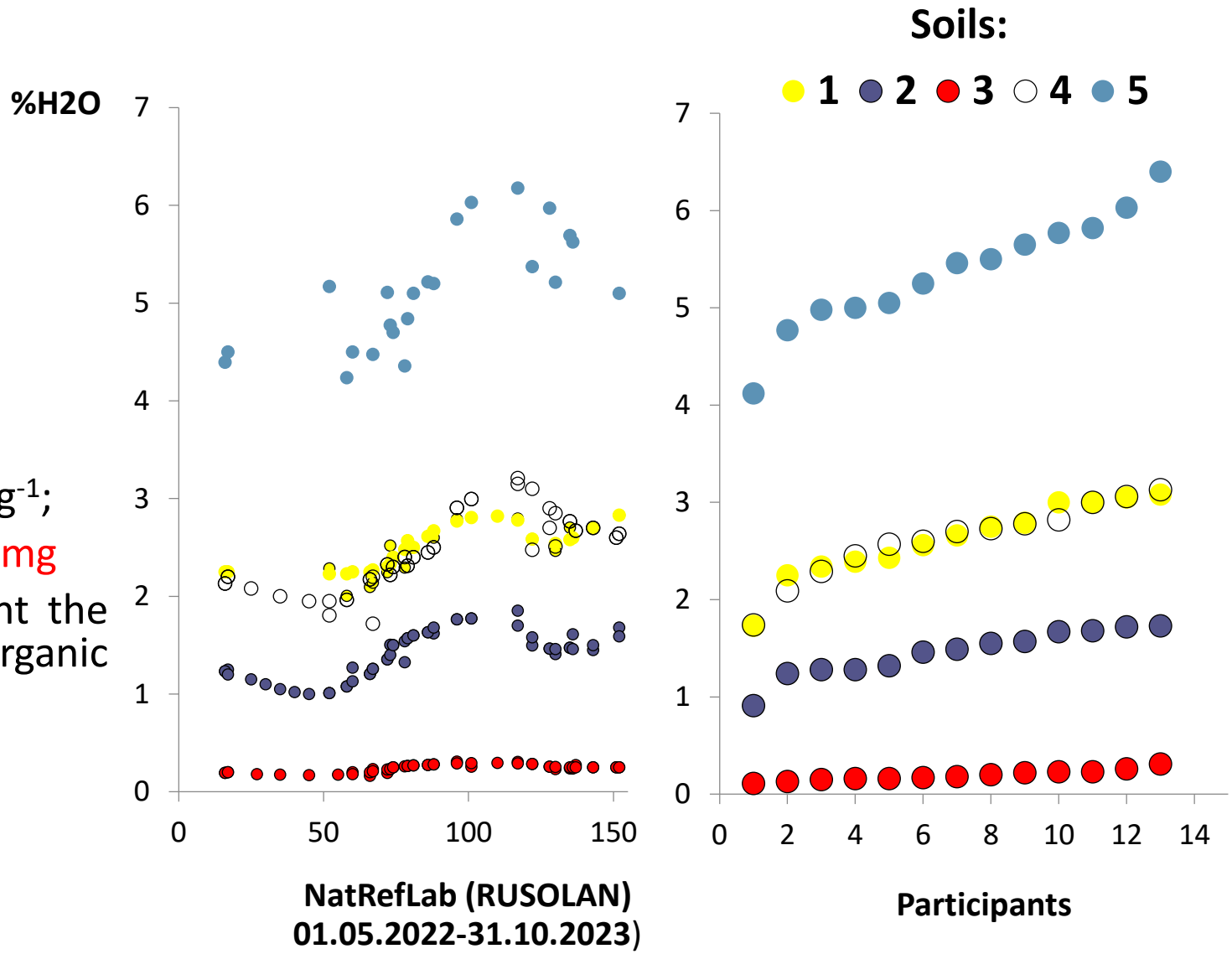


Soil moisture (*W*)

$$\%SOC = \frac{(A - A_0)}{Km} 100$$

- A – absorbance of study solution;
- A_0 – coefficient of calibration function;
- K – coefficient of calibration function, mg^{-1};
- m – mass of tested soil (***dried at 105˚C***), mg
- f – correction factor taking into account the incomplete oxidation of carbon of organic compounds

$$W = \frac{m_{\text{air dry soil}}}{m_{\text{dried at 105°C}}}$$



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Dry combustion on the analyzer

Dry combustion	–	+	+	–	+	–	–	–	–	+	+	+	–	+	–	+	–	+	–	–	+	+	–	–	–	–	–	–	**11**
LOI	+	+	–	–	–	–	–	–	+	+	+	+	+	+	+	+	–	+	–	–	–	+	–	+	–	–	–	+	**14**
Tyurin's method	+	+	+	+	–	–	+	+	–	+	+	+	+	+	+	–	+	+	–	+	–	+	+	+	–	+	–	+	**20**
Walkley-Black method	+	+	–		–	–	+	–	–	+	+	+	+	+	–	–	–	+	–	–	+	+		+	–	–	–	+	**13**
Number of laboratory	Lab_1	Lab_2	Lab_3	Lab_3	Lab_4	Lab_5	Lab_6	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_20	Lab_21	Lab_22	Lab_23	Lab_24	Lab_25		

Analyzers:

- EA 1110 (CHNS-O)
- CHNS/O PE2400-II (анализ Прегла-Думаса)
- HCNS-анализатор Elementar Vario Cube
- автоматический анализатор общего органического углерода – ENVIRO TOC
- анализатор углерода и серы Метавак-CS.





Dry combustion on the analyzer

Values of uncertainty (error) of measurement results (by: МИ 88-17641-004-2016)

Mass fraction measurement range, ω($C_{орг}$), %	Uncertainty (accuracy) indicator (P = 0,95), $U_{АВ\ отн}$, % (±$\delta_л$, %)
0,1-2	23
2-5	15
5-10	10

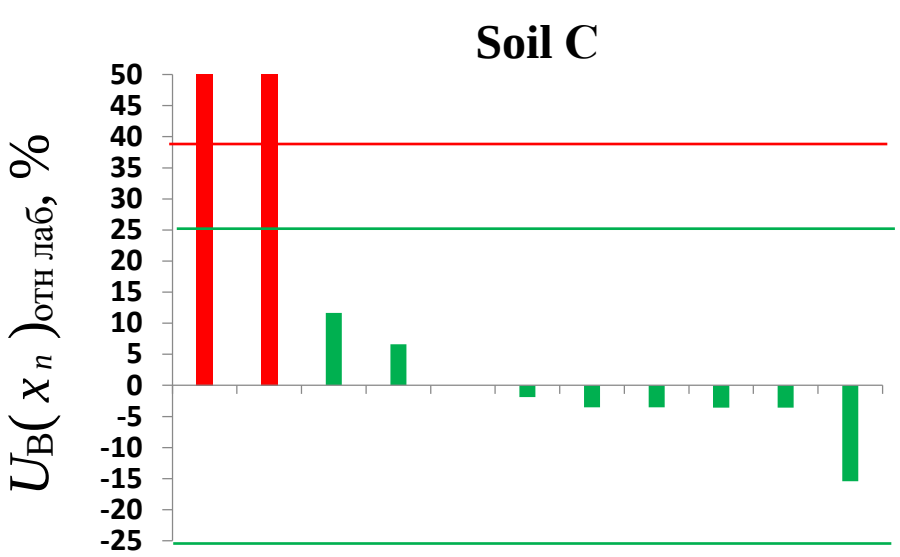
Warning and action limits (lower and upper)

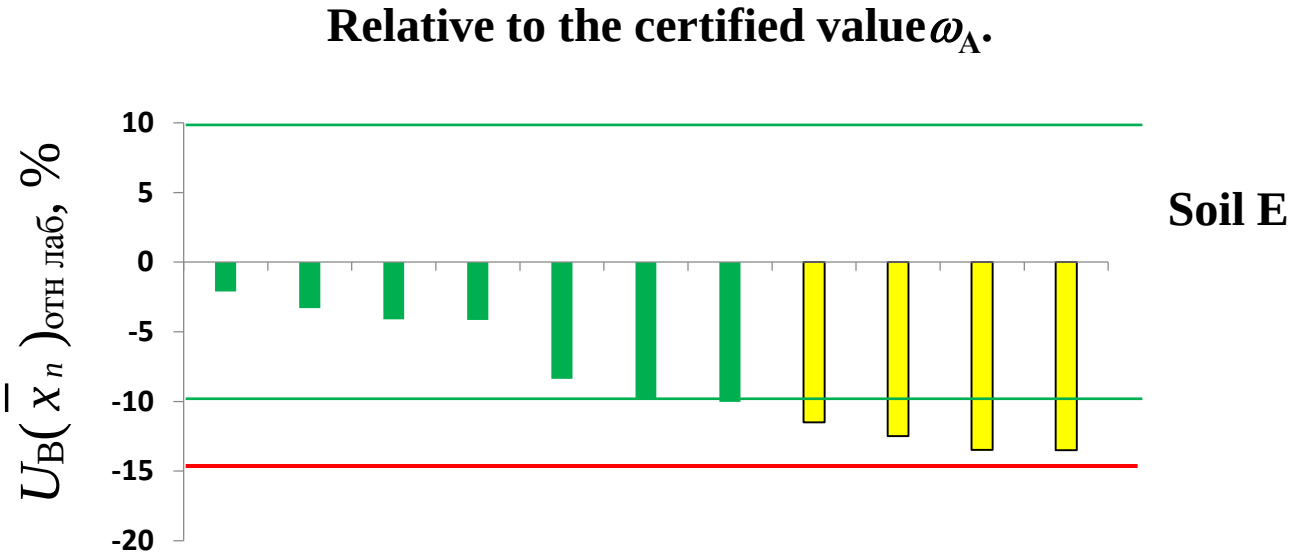
Control soils	ω_A, %	$\omega_A - U_{АВ}(\omega_A)_{абс}$	$\omega_A + U_{АВ}(\omega_A)_{абс}$	$\omega_A - 1{,}5U_{АВ}(\omega_A)_{абс}$	$\omega_A + 1{,}5U_{АВ}(\omega_A)_{абс}$
Soil A (F, I)	**6,44**	5,80	7,08	5,47	7,41
Soil B (G, H)	**2,32**	1,97	2,67	1,80	2,84
Soil C	**0,197**	0,15	0,24	0,13	0,26
Soil D	**0,12**	0,09	0,15	0,08	0,16
Soil E	**7,87**	7,08	8,66	6,69	9,05

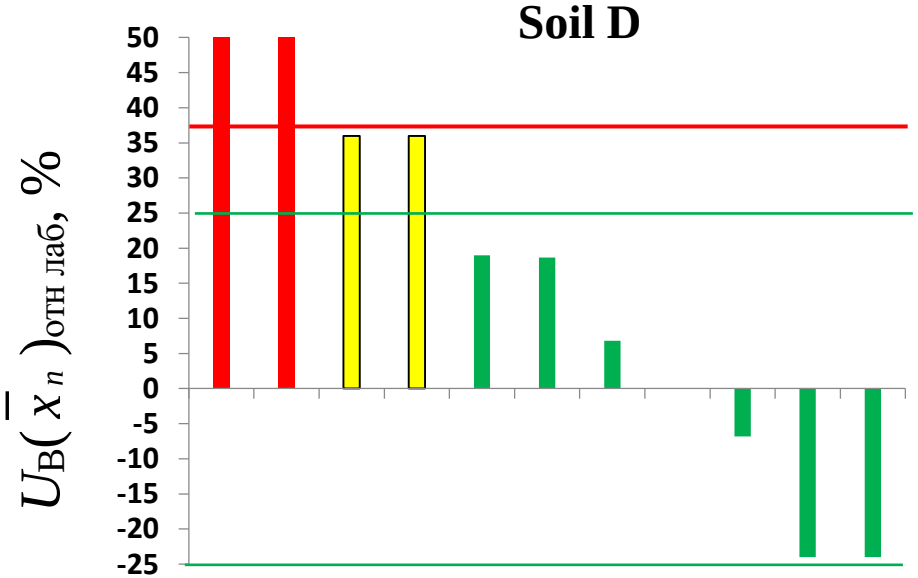
	Satisfactory result – в пределах предупреждения: $\lvert x_л - \omega_A \rvert \le U_{АВ}(\omega_A)_{абс}$.
	Doubtful result– в пределах действия: $U_{АВ}(\omega_A)_{абс} < \le 1{,}5\ U_{АВ}(\omega_A)_{абс}$. It is necessary to repeat the procedures of the method.
	Unsatisfactory result (miss) – за пределами действия: $> 1{,}5\ U_{АВ}(\omega_A)_{абс}$.

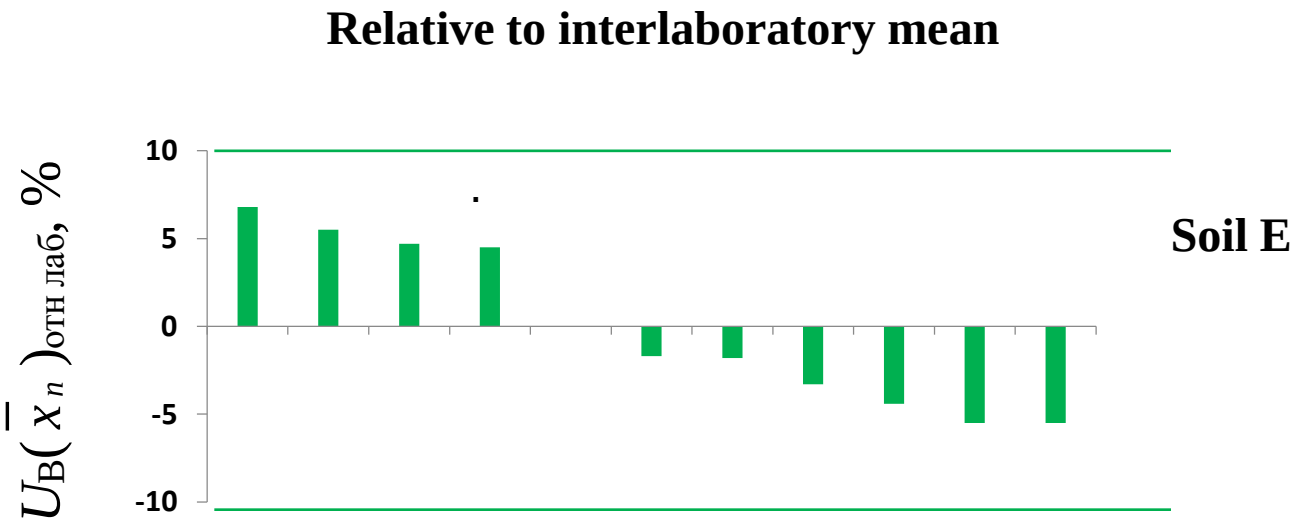
Number of laboratory	Control soils				
	A F I	B G H	C	D	E
Lab_1	–	–	–	–	–
Lab_2	6,3	2,27	0,21	0,16	7,21
Lab_3	5,82	2,02	0,39	0,19	6,81
Lab_3					
Lab_4	6,66	2,49	0,22	0,16	7,55
Lab_5	–	–	–	–	–
Lab_6	–	–	–	–	–
Lab_6					
Lab_7	–	–	–	–	–
Lab_8	6,69	2,36	0,19	0,09	7,7
Lab_9	6,41	2,34	0,19	0,11	7,54
Lab_10	5,77	1,95	0,17	0,09	6,89
Lab_11	–	–	–	–	–
Lab_12	5,97	2,14	0,19	0,14	7,08
Lab_13	–	–	–	–	–
Lab_14	6,18	2,25	0,19	0,11	6,97
Lab_15	–	–	–	–	–
Lab_16	6,35	2,23	0,19	0,14	7,09
Lab_17	–	–	–	–	–
Lab_18	–	–	–	–	–
Lab_19	6,64	2,44	0,2	0,12	7,61
Lab_20	6,08	2,21	0,44	0,41	6,81
Lab_20					
Lab_21	–	–	–	–	–
Lab_22	–	–	–	–	–
Lab_23	–	–	–	–	–
Lab_24	–	–	–	–	–
Lab_25	–	–	–	–	–

Dry combustion on the analyzer









Results of interlaboratory comparison tests using the dry combustion method

Control soils	Soil sample		Mass fraction C_{orr}	Expanded uncertainty type A (absolute)	Absolute divergence	Boundary of significance of displace-ment (absolute)	Number of results: total/ miss	Relative displacement
	min	max	$\overline{\overline{\omega}}$, %	$U_A(\overline{\overline{\omega}})_{абс}$, %	$\lvert\overline{\overline{\omega}} - \omega_A\rvert$, %	$\theta(\Delta)$, %	N/N^*	$\theta(\delta)$, %
Soil A	0,0009	0,40	**6,26**	0,20	-0,18	0,20	11	**-2,8**
Soil B	0,0009	0,56	**2,25**	0,10	-0,07	0,10	11	**-3**
Soil C	0,0010	0,65	**0,19**	0,01	-0,01	0,01	11/2*	**-5**
Soil D	0,0010	0,54	**0,12**	0,02	0	0,02	11/2*	**0**
Soil E	0,0010	0,31	**7,20**	0,20	**-0,67**	0,21	11	**-8**

Loss-on-ignition method

Gravimetric methods of analysis

Conditions : T 800-1100 °C; $m_{всп}$ 2-25 g; t 1-5,5 h (to a constant mass within 1 hour **)**

GOST* 26801-86 Peat. Method for determination of ash content in deposit (https://rags.ru/gosts/gost/38895/)

GOST 11306-2013 Peat and products of its processing. Methods for determination of ash content (https://rags.ru/gosts/gost/56090/)

Vorobieva L.A. Theory and practice of chemical analysis of soils. Moscow. 2006. 400P.

Luganskaya V.D., Lugansk V.N. Chemical analysis of soils. Ekaterenburg. 2011

Samofalova I.A., Lobanova E.S. Soil science. Permian. 2021.

Conditions : T 525-550 °C; $m_{всп}$ 2-20 g; t 1-4,5 h (to a constant mass within 1 hour **)**

GOST 23740-2016 Soils. Methods of laboratory determination of organic composition (https://rags.ru/gosts/gost/64068/)

Conditions : preliminary removal of chlorides and carbonates , t 525±25°C; $m_{всп}$ от 20g; T to constant mass .

GOST 27784-88 Soils. Method for determination of ash content in peat and peat-containing soil horizonts (https://rags.ru/gosts/gost/11647/)

Conditions : T 525±25°C; $m_{всп}$ 10-15g; t 4,5 h (to constant weight within an hour)

GOST P 57065-2016 Resources saving. Waste treatment. Determination of loss on ignition in waste, sludge and sediments (https://rags.ru/gosts/gost/62909/)

Conditions : T 550±25°C; $m_{всп}$ не более 5 g; t 1 h (to constant weight within an hour)

GOST 11306-2013 Peat and products of its processing. Methods for determination of ash content (https://rags.ru/gosts/gost/56090/)

Conditions : T 550±25°C; $m_{всп}$ не более 2-8 g; t 1 h (to constant weight within an hour)

*Russian state standard

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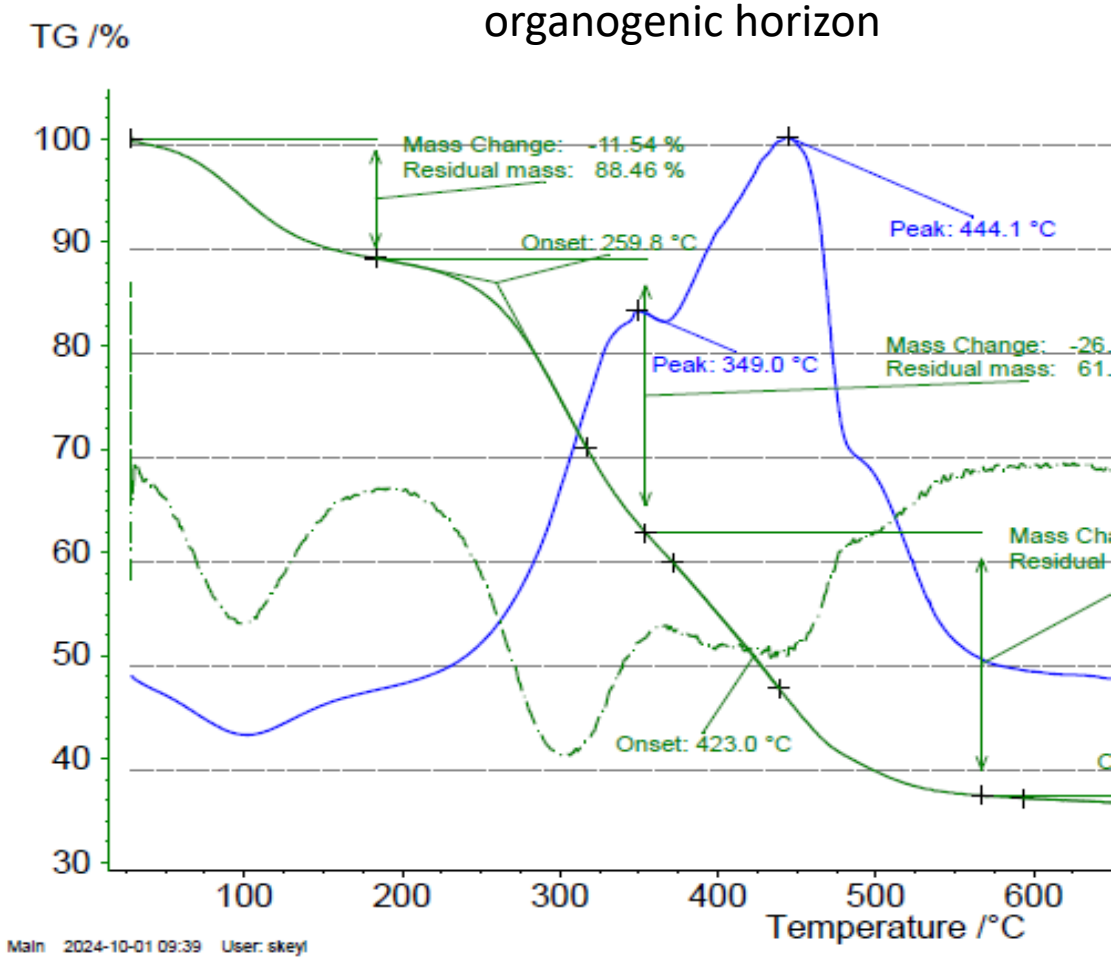
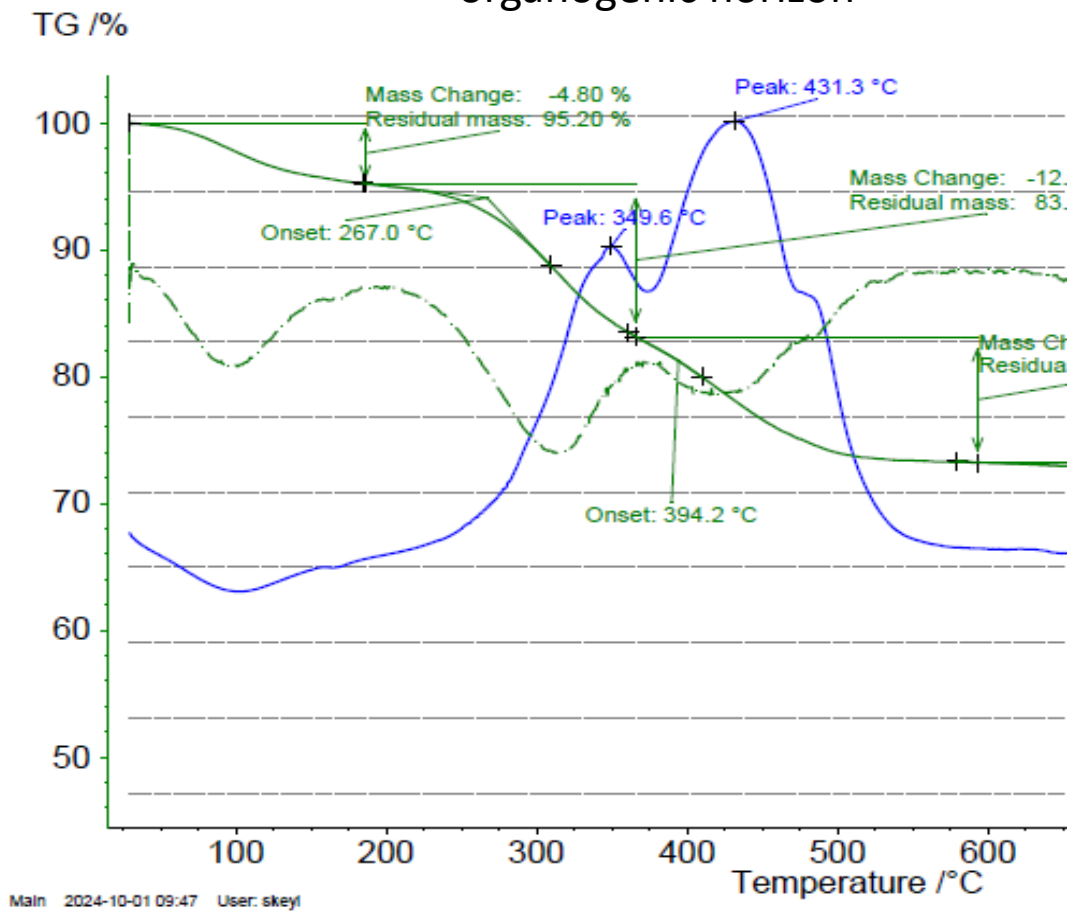
LOI: temperature - choice of conditions

organogenic horizon



organogenic horizon

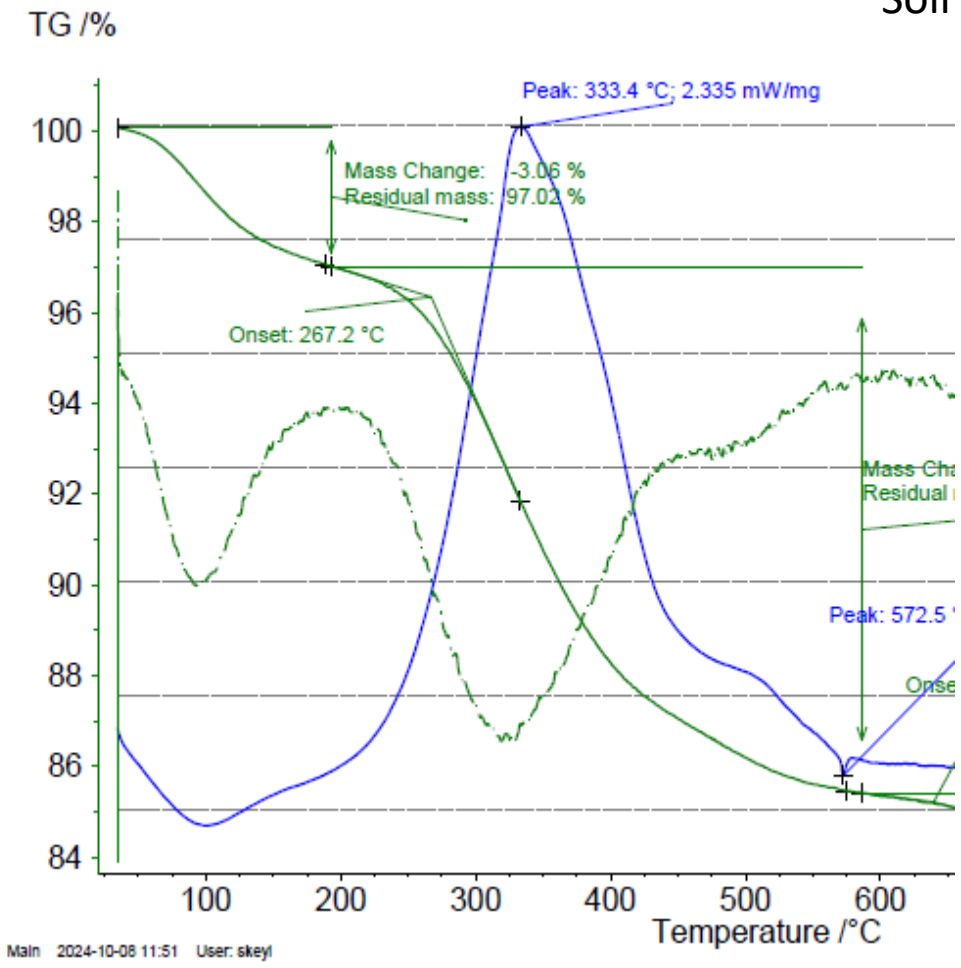


$\omega(\text{ппп})_{\text{ТГ}} \approx 1{,}724\ \omega(\text{C}_{\text{орг}}),\ \ \alpha(\text{C}_{\text{орг}}) = 0{,}58$

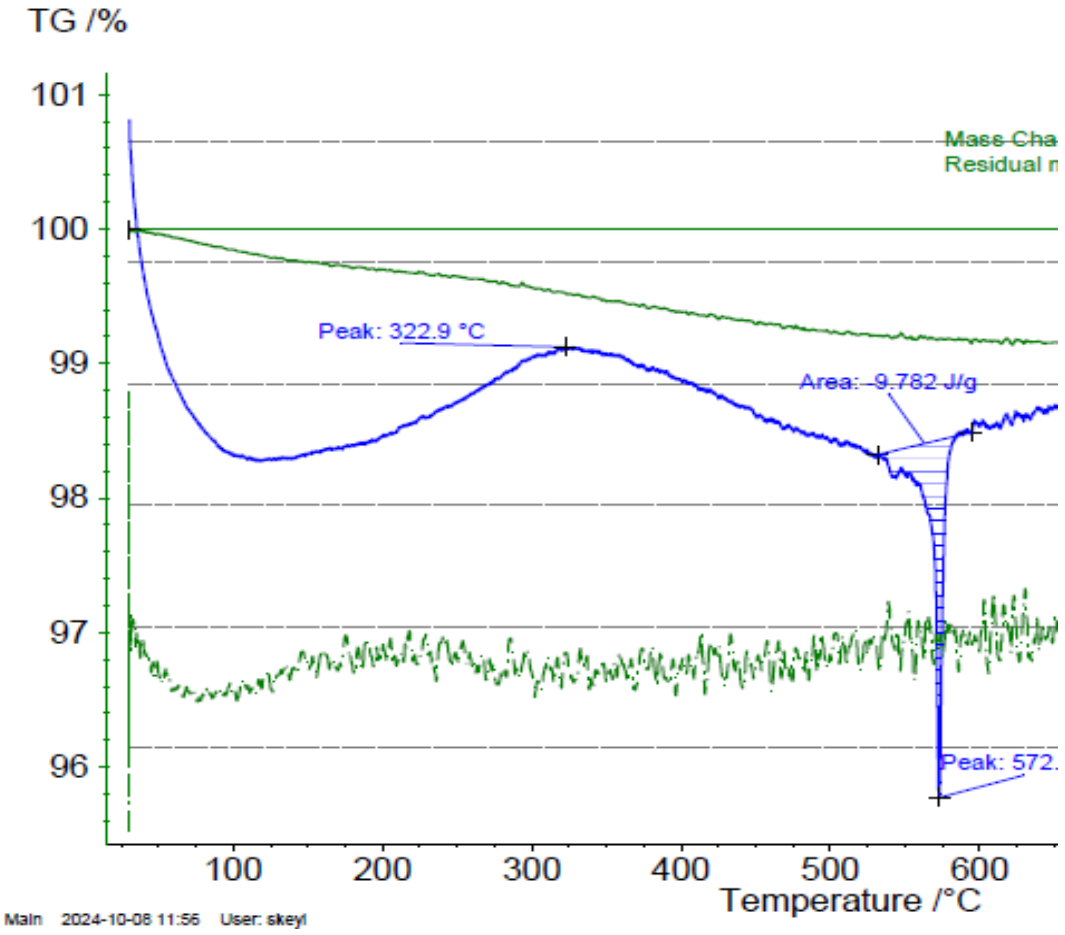
T= 550 °C

Loss-on-ignition method

Soil A



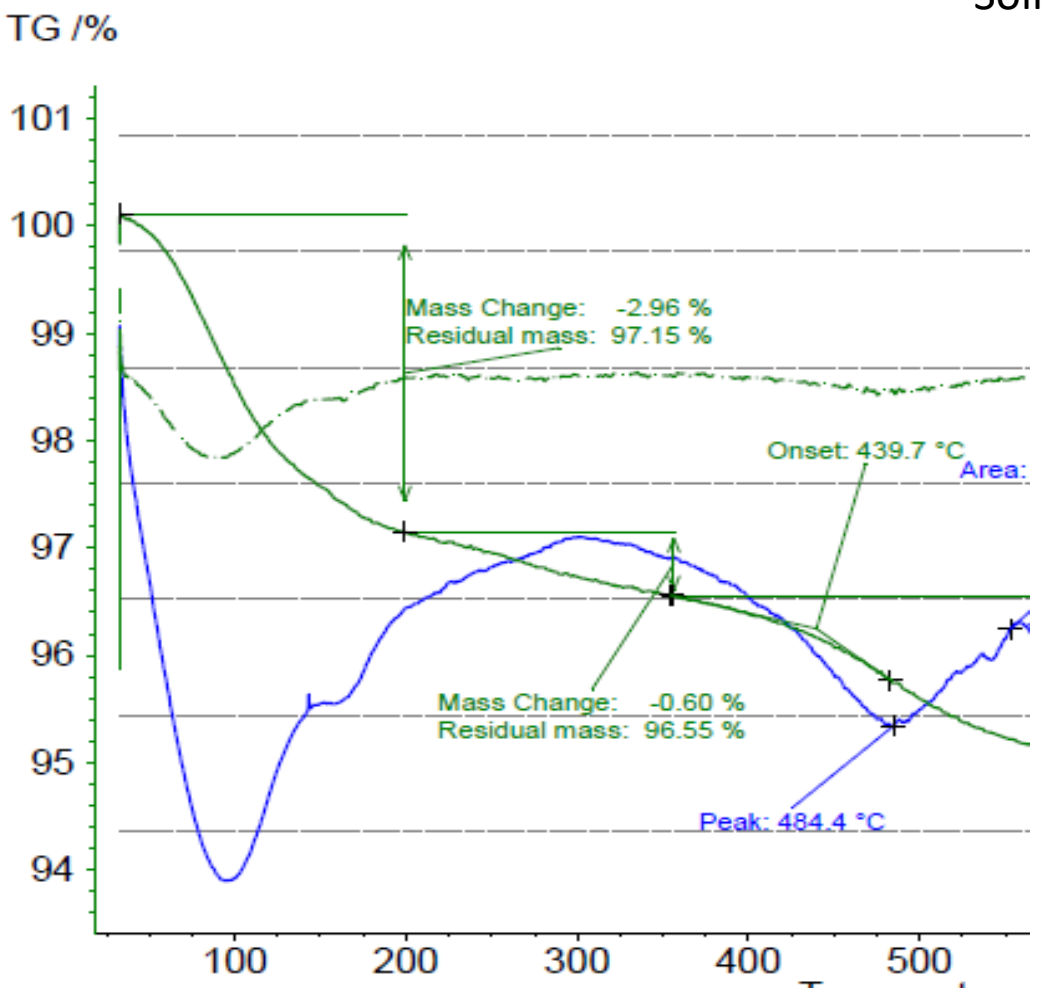
Soil C



T= 550 °C

Loss-on-ignition method

Soil D



T= 550 °C

LOI: Heating time – choice of conditions

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	16,8390	13,1658	13,1656	13,1656	13,1656	13,1654	13,1650

t = 7 hours

Loss-on-ignition method

Soil type	Dry combustion on the analyzer	Loss-on-ignition method			
	$\omega(C_{орг})$, %	ω(LOI), %	$U_A(\omega(LOI))_{абс}$, %	$U_A(\omega(LOI))_{отн}$, %	**ω(LOI) · 0,58**
Soil A (F, I)	**6,44**	**12.9**	0,10	0.80	**7,48**
Soil B (G, H)	**2,32**	**5.45**	0,18	3.3	**3,16**
Soil C	**0,197**	**0.66**	0,05	8.2	**0,38**
Soil D	**0,118**	**2.77**	0,22	8.2	**1,61**
Soil E	**7,87**	**19.0**	0,34	1.8	**11,02**

Loss-on-ignition method

Values of uncertainty (error) of measurement results

Mass fraction measurement range, ω(LOI), %	Uncertainty (accuracy) indicator (P = 0,95), $U_{AB\ отн}$, % ($\pm\delta_л$, %)
0,5-5	25
5-10	10
10-25	5

Warning and action limits (lower and upper)

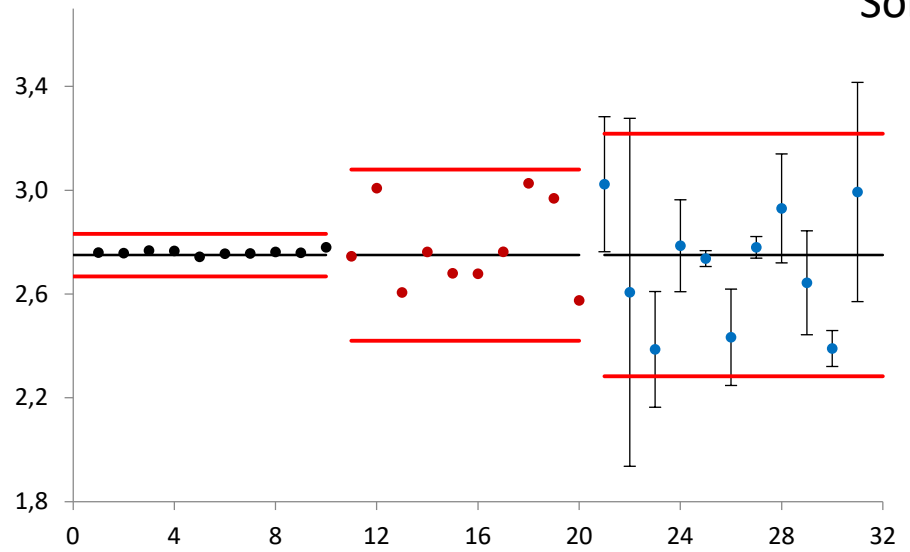
Control soils	ω_A, %	$\omega_A - U_{AB}(\omega_A)_{абс}$	$\omega_A + U_{AB}(\omega_A)_{абс}$	$\omega_A - 1{,}5U_{AB}(\omega_A)_{абс}$	$\omega_A + 1{,}5U_{AB}(\omega_A)_{абс}$
Soil A (F, I)	**12.9**	12,3	13,5	11,9	13,9
Soil B (G, H)	**5.45**	4,91	6,00	4,63	6,27
Soil C	**0.66**	0,49	0,82	0,41	0,90
Soil D	**2.77**	2,08	3,46	1,73	3,81
Soil E	**19.0**	18,1	20,0	17,6	20,4

	Satisfactory result – в пределах предупреждения: $\lvert x_л - \omega_A \rvert \le U_{AB}(\omega_A)_{абс}$.
	Doubtful result– в пределах действия: $U_{AB}(\omega_A)_{абс} < \le 1{,}5\ U_{AB}(\omega_A)_{абс}$. It is necessary to repeat the procedures of the method.
	Unsatisfactory result (miss) – за пределами действия: $> 1{,}5\ U_{AB}(\omega_A)_{абс}$.

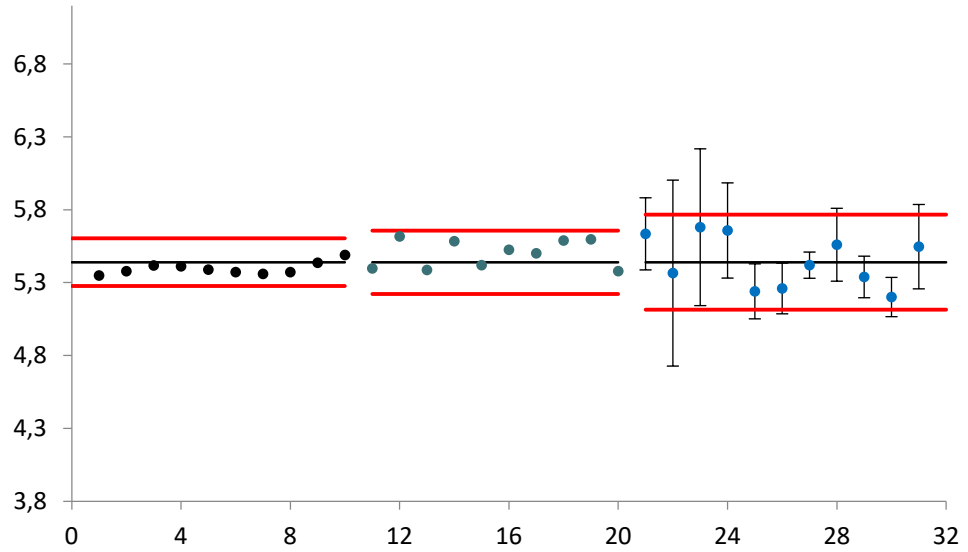
Number of laboratory	Control soils				
	A F I	B G H	C	D	E
Lab_1	13,0	5,63	0,65	3,02	19,2
Lab_2	12,8	5,37	0,76	2,61	17,7
Lab_3	–	–	–	–	–
Lab_3					
Lab_4	–	–	–	–	–
Lab_5	–	–	–	–	–
Lab_6	–	–	–	–	–
Lab_6					
Lab_7	12,8	5,39	0,28	1,88	19,0
Lab_8	13,2	5,68	0,72	2,39	18,8
Lab_9	13,3	5,66	0,61	2,79	18,8
Lab_10	12,6	5,24	0,64	2,74	19,0
Lab_11	12,6	5,26	0,58	2,43	18,1
Lab_12	12,9	5,42	0,63	2,78	19,3
Lab_13	15,0	6,77	0,76	5,04	22,8
Lab_14	13,0	5,56	0,70	2,93	19,2
Lab_15	–	–	–	–	–
Lab_16	12,6	5,47	0,59	2,55	18,3
Lab_17	–	–	–	–	–
Lab_18	–	–	–	–	–
Lab_19	–	–	–	–	–
Lab_20	12,7	5,34	0,62	2,64	18,7
Lab_20					
Lab_21	12,5	5,20	0,59	2,39	18,5
Lab_22	–	–	–	–	–
Lab_23	–	–	–	–	–
Lab_24	–	–	–	–	–
Lab_25	12,9	5,55	0,65	2,99	19,5

Loss-on-ignition method

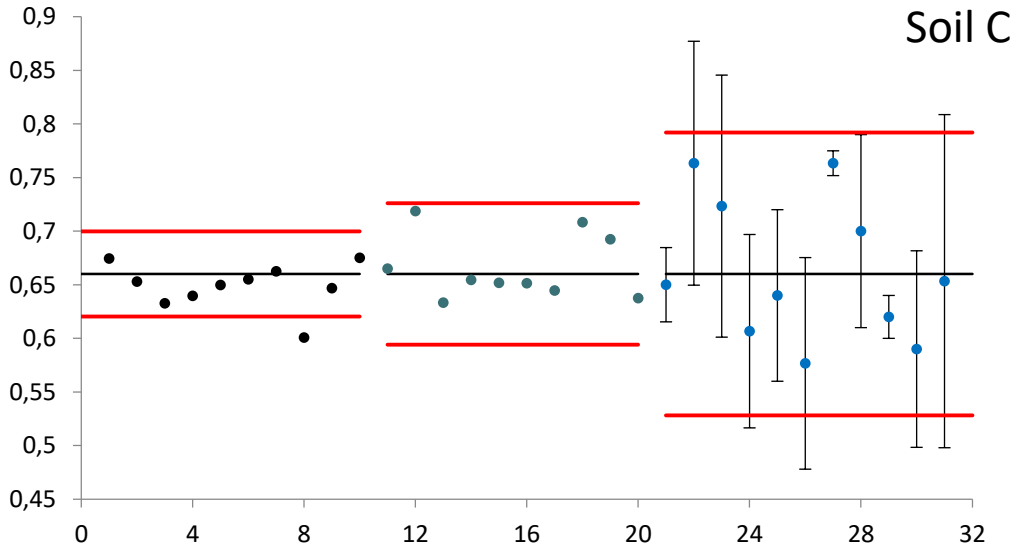
Soil D



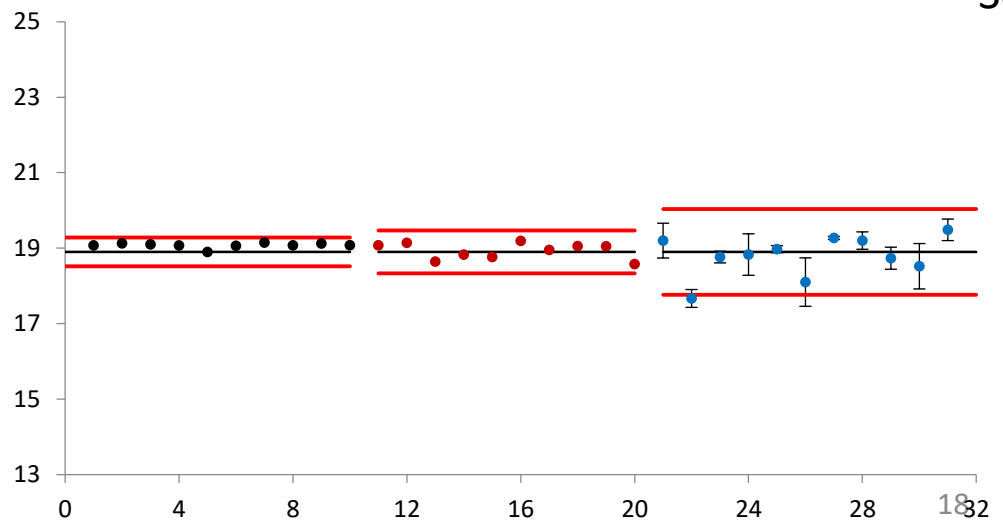
Soil B



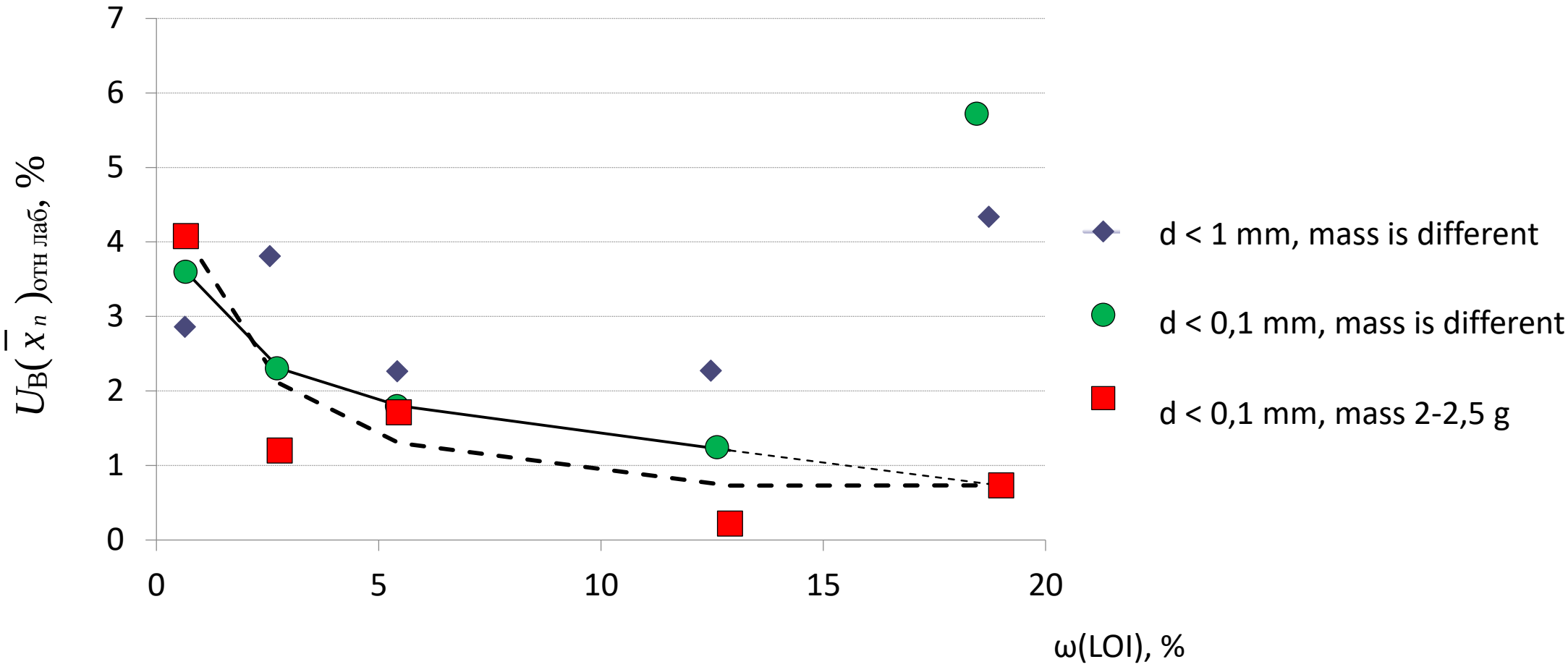
Soil C



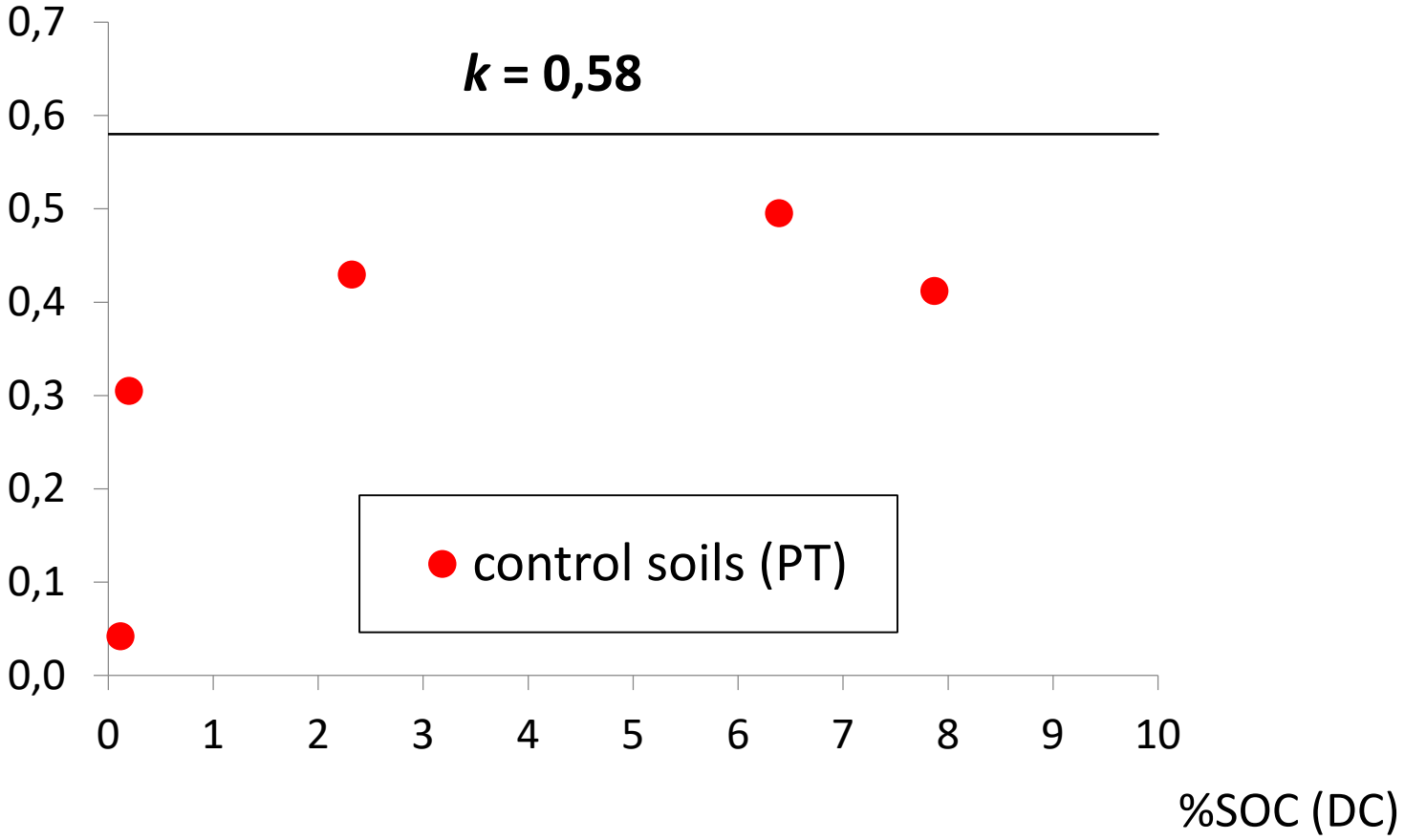
Soil E



Loss-on-ignition method



***k* = %SOC(DC) / %LOI**



In the literature ***k* = 0.4-0.71** (Kamara et al., 2007; Pribyl, 2010; Roper et al., 2019).

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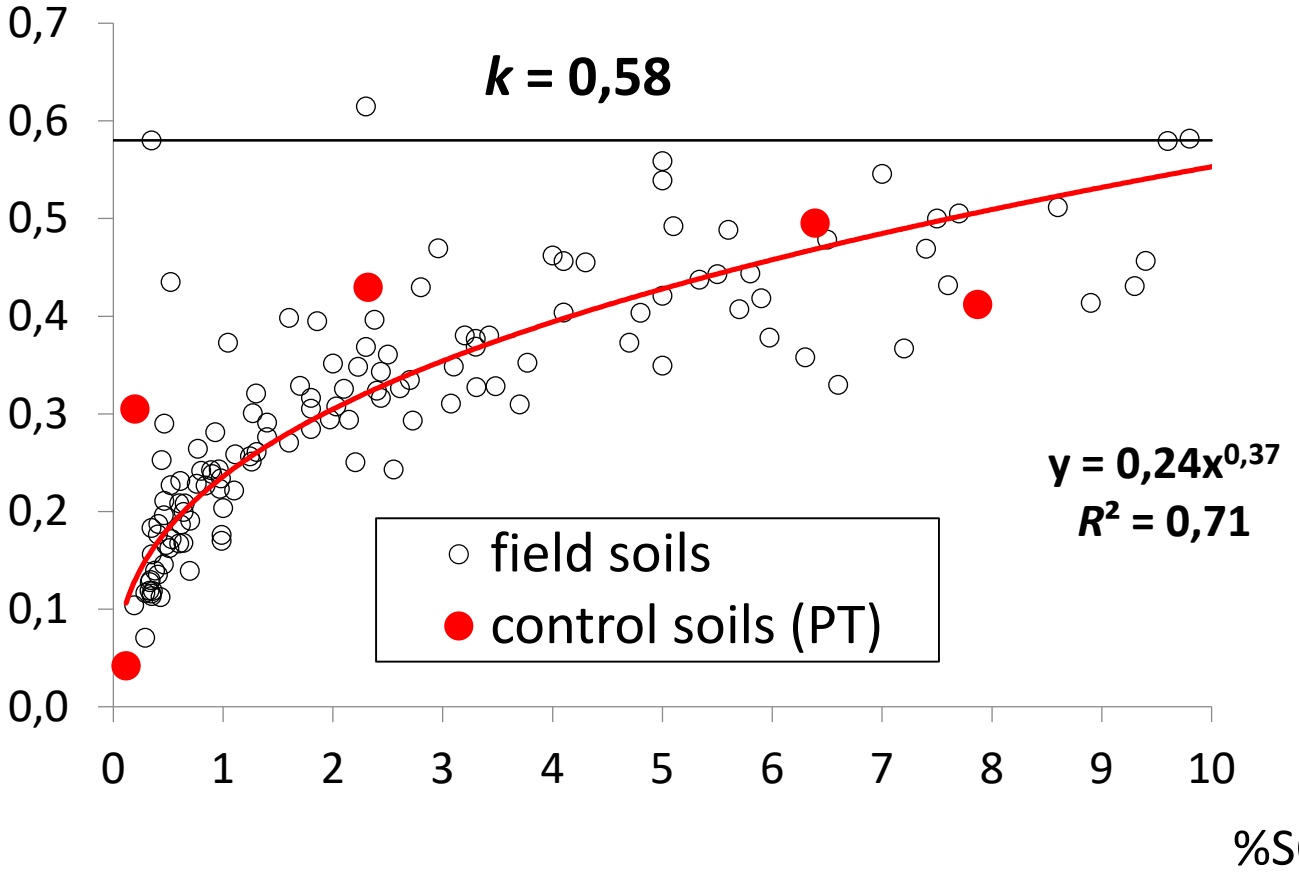
***k* = %SOC(DC) / %LOI**



In the literature ***k* = 0.4-0.71** (Kamara et al., 2007; Pribyl, 2010; Roper et al., 2019).

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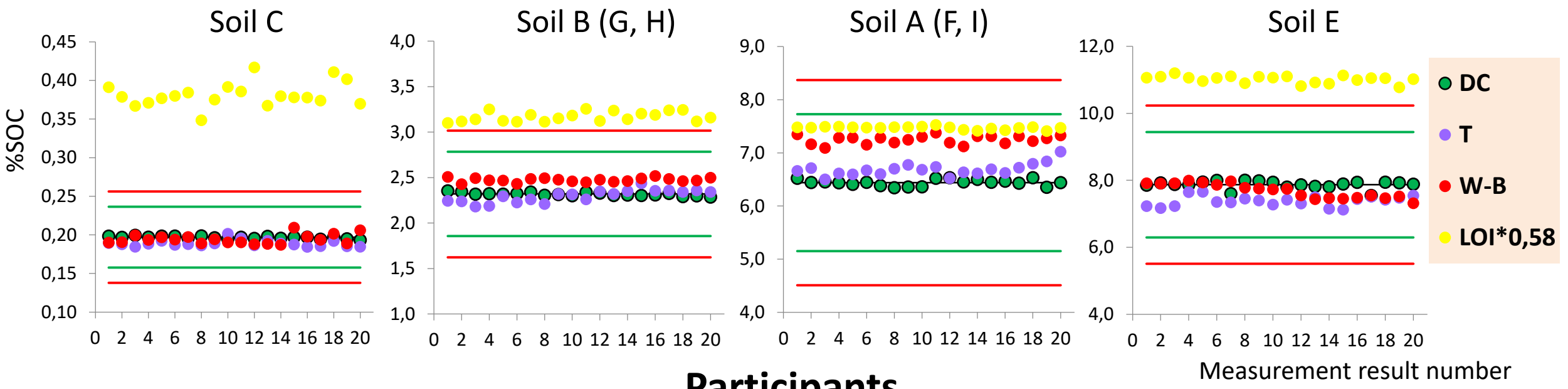
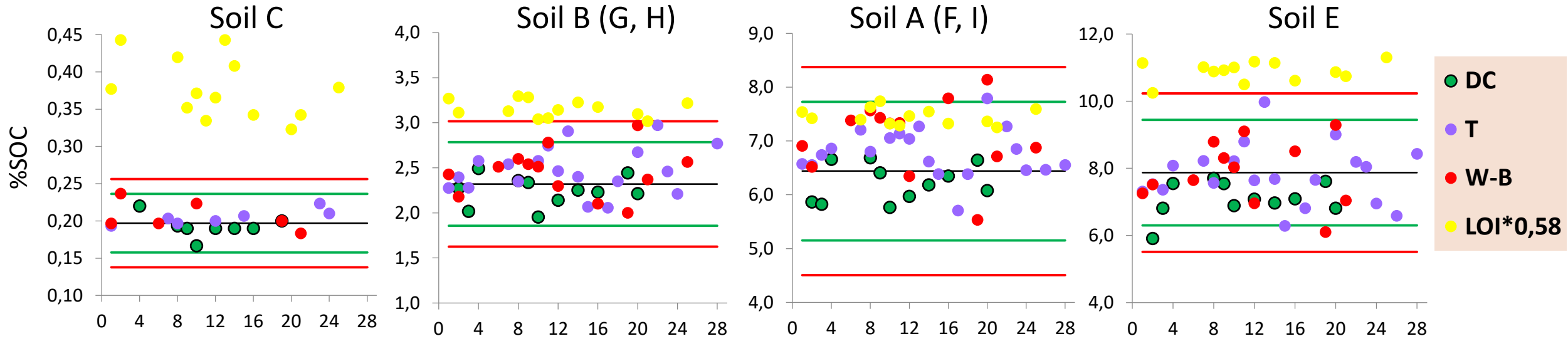
NatRefLab (RUSOLAN)

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



Participants



Measurement result number / *not a laboratory number!!!!!!*

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

Thank you for attention!

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