Determination of Particle Size Distribution in Central Forest Laboratory

National Forest Centre in Zvolen

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Determination of Particle Size Distribution

Plan of presentation

- Treatment of the samples
  - Removal of C-org.
  - Destruction of $CaCO_3$
  - Checking EC
- Dispersion and Separation
- Calculation
- BioSoil samples
- QA/QC

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Determination of Particle Size Distribution

Weighing of the samples

- weigh 10 g of dried soil
- in 50 ml beaker for the samples without treatment
- in 250 (600) ml glass beaker for the samples with removal of organic matter and carbonate

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Determination of Particle Size Distribution

Destruction of Organic Carbon

- we remove organic matter in the samples containing more than five percent mass fraction
- add 30 ml distilled water and 30 ml of the 30% hydrogen peroxide solution

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Destruction of Organic Carbon

- check, stir frequently, the samples can make intensive foam
- check foaming with ethanol
- be careful you can lost some mass of samples by foam
- cover and leave overnight

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Destruction of Organic Carbon

- the next day place the vessels in thermo regulated bath
- heat to 95°C and keep during 2 hours, checking and stir
- refill water
- cool to 50°C and take out
- add 25 ml 1M Calcium chloride

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Destruction of Organic Carbon

- refill water to 250 ml
- let stand until the supernatant is clear

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Determination of Particle Size Distribution

Destruction of Organic Carbon

- siphon off the supernatant - tip of pipette about 5 - 10 mm above the deposition

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Determination of Particle Size Distribution

Removal of Carbonate

- more than 2 % mass fraction
- add chlorid acid calculated according the content of carbonates
- add water at least to volume 250 ml
- place into in thermo regulated water bath for 15 minutes at about 80 C

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Determination of Particle Size Distribution

Removal of Carbonate

- suspension to stand overnight
- to leave a perfectly clear supernatant
- this can be siphoned off
- add water – removal hydrochloric acid
- check EC – repeat washing less than 40 ms/m

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Determination of Particle Size Distribution

Dispersion

- add dispersing agent, stir
- leave the suspension to stand overnight
- next day place into ultrasonic bath for 4 minutes

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Determination of Particle Size Distribution

Separation of fractions

- transfer the dispersed suspension from beaker quantitatively onto the 63 um sieve
- wash the soil using jet of water from wash bottle until the water runs clear
- to sieve into 1000 ml glass sedimentation cylinders

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Determination of Particle Size Distribution

Separation of fractions

- remove the sand from sieve into Petri glass dish
- wash the bottom of sieve into the sedimentation tube
- place Petri glass dish in an oven between 105 and 110 °C

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Determination of Particle Size Distribution

Separation of fractions

- fill out sedimentation tube by water and agitate at least 30 times for 2 minutes
- leave to sediment according determined time
- samples are taken after 1 minute in the depth of 15 cm and in 3 hours and 47 minutes in the depth of 5 cm from sedimentation cylinder at temperature 21°C

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Determination of Particle Size Distribution

Separation of fractions

- drain the fractions and wash pipette into a weighing vessel of know mass
- 1st fraction clay and silt
- 2nd fraction clay

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Determination of Particle Size Distribution

Separation of fractions

- the placement of different fractions in an oven
- each fraction has its own stage in oven

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Determination of Particle Size Distribution

Separation of fractions

- drying fractions cooled in the desiccators
- the results of weighing are written down on the formula paper
- rewrite in the computer
- excel file make automatic calculation the percentage part of fractions

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Determination of Particle Size Distribution

Primary writing of the results – in the EXCEL File

TLAČIŤ				**Stanovenie zrnitostného zloženia pôd**											**ŠAP 5.**		
Protokol:				Číslo úlohy:					**Dátum:**		**Spracoval:**					Teplota:	
vzorce						Fx - Ex	**Gx*100/10**				Kx - Jx	**(Lx-Qx)*100/10*1000/25**				Px - Ox	**(Qx-bl)*100/10*1000/25**
Číslo vzorky		Hĺbka	**PIESOK**					**PRACH**					**ÍL**				
	C-org	**Ca-CO3**	číslo misky	hmotnosť misky	hmotnosť frakcie + misky	hmotnosť frakcie	frakcia	číslo misky	hmotnosť misky	hmotnosť frakcie + misky	hmotnosť frakcie	frakcia	číslo misky	hmotnosť misky	hmotnosť frakcie + misky	hmotnosť frakcie	frakcia
	%	**%**		**g**	**g**	**g**	**%**		**g**	**g**	**g**	**%**		**g**	**g**	**g**	**%**
						0,0000	0,00				0,0000	0,00				0,0000	-8,00
						0,0000	0,00				0,0000	0,00				0,0000	-8,00
						0,0000	0,00				0,0000	0,00				0,0000	-8,00
						0,0000	0,00				0,0000	0,00				0,0000	-8,00
						0,0000	0,00				0,0000	0,00				0,0000	-8,00
						0,0000	0,00				0,0000	0,00				0,0000	-8,00
						0,0000	0,00				0,0000	0,00				0,0000	-8,00

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Determination of Particle Size Distribution

This formulas are in Excel cell for automatic calculation

Calculation:

Clay in % = $(m_3 - m_r)$. f 1
Silt in % = $(m_2 - m_3)$. f 1
Sand in % = m_1 . f 2

$\mathbf{m_3}$ – fraction clay mass
$\mathbf{m_2}$ – fraction silt + clay mass
$\mathbf{m_1}$ – fraction sand mass
$\mathbf{m_r}$ – dispersing agent mass - g (0,02 g for 25 ml pipette)
$\mathbf{V_c}$ – pipette volume - ml (25ml)
m – sample mass – g (10g)
V – cylinder volume – ml (1000ml)
f 1 – 100 / **m** * **V** / $\mathbf{V_c}$ (f 1 = 100/ 10g * 1000ml / 25ml = 400)
f 2 – 100 / **m** **100/10=10**

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Determination of Particle Size Distribution

Quality check

Clay + silt + sand + C-org. + $CaCO_3$ + (100-DM) = 100 %

Plot	horizon	> 63 um	2 - 63 um	< 2 um	SUM of	Corg.	CaCO3	100-DM	Total
		%	%	%	fractions	%	%	%	%
A 10	H7	46,9	31,3	20,4	99	0,3	0,0	2,0	101
B 7	H5	96,6	0,2	4,4	101	0,3	0,0	0,2	102
Y4	H5	5,8	58,6	32,6	97	0,7	0,0	2,3	100
R 6	H1	18,1	50,9	16,1	85	10,4	0,0	4,8	100
R 2	H1	3,0	56,9	19,0	79	16,3	0,0	7,8	103
P 6	H2	53,1	26,0	12,3	91	7,1	0,0	2,3	101
C 10	H4	71,5	6,7	5,0	83	0,5	15,6	0,4	100
C 10	H5	40,9	20,7	9,7	71	0,0	28,0	1,0	100
H3	H3	0,6	15,8	19,5	36	0,6	62,0	2,5	101
E7	H2	1,5	45,2	12,2	59	5,7	32,0	6,1	103
K6	H3	6,7	6,7	25,8	39	5,9	52,0	5,9	103
O 5	H3	2,2	24,1	26,5	53	5,4	38,0	3,6	100

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Determination of Particle Size Distribution

Quality check

Clay + silt + sand + C-org. + $CaCO_3$ + (100-DM) = 100 %

sample	sample		> 63 um	2 - 63 um	< 2 um	Sum	Corg.	CaCO3	100-DM	Total
	plot	horizon	%	%	%	%	%	%	%	%
06-5806	O 5	H 4	2,7	10,5	5,9	19	0,78	54	0,57	74
			2,7	10,5	5,9	19	0,78	69	0,57	89
			2,7	10,5	5,9	19	0,78	77	0,57	97
			2,7	10,5	5,9	19	0,78	84	0,57	104
			2,7	10,5	5,9	19	0,78	87	0,57	107
	average		2,7	10,5	5,9	19	0,78	74	0,57	95
06-6148	C 7	H 5	0,2	0,1	9,8	10	4,71	49	0,62	64
			0,2	0,1	9,8	10	4,71	58	0,62	73
			0,2	0,1	9,8	10	4,71	60	0,62	75
			0,2	0,1	9,8	10	4,71	66	0,62	81
			0,2	0,1	9,8	10	4,71	75	0,62	90
average			0,15	0,1	9,8	10,05	4,71	62	0,62	77

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Determination of Particle Size Distribution

Recalculation for mineral soil

Clay + silt + sand = 100 %

Plot	horizon	> 63 um	2 - 63 um	< 2 um	sum of		> 63 um	2 - 63 um	< 2 um	Total
		%	%	%	fractions		%	%	%	recalculation
A 10	H7	46,9	31,3	20,4	99		47,6	31,8	20,7	100
B 7	H5	96,6	0,2	4,4	101		95,4	0,2	4,3	100
Y4	H5	5,8	58,6	32,6	97		6,0	60,4	33,6	100
R 6	H1	18,1	50,9	16,1	85		21,3	59,8	18,9	100
R 2	H1	3,0	56,9	19,0	79		3,9	72,1	24,0	100
P 6	H2	53,1	26,0	12,3	91		58,1	28,4	13,5	100
C 10	H4	71,5	6,7	5,0	83		86,0	8,0	6,0	100
C 10	H5	40,9	20,7	9,7	71		57,4	29,0	13,6	100
H3	H3	0,6	15,8	19,5	36		1,8	44,1	54,2	100
E 7	H2	1,5	45,2	12,2	59		2,5	76,8	20,7	100
K 6	H3	6,7	6,7	25,8	39		17,2	17,1	65,7	100
O 5	H3	2,2	24,1	26,5	53		4,1	45,6	50,3	100

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BioSoil samples

Number of samples		Total	without treatment	Removal		
				CaCO3	C-org.	CacO3+C-org
I. level	total	868	631	118	68	51
	fix depht	435	305	28	72	30
	diagnostic horizon	433	326	40	46	21
II. level	fix depht	108	98	0	10	0

fraction		> 63 um	2 - 63 um	< 2 um
unit		%	%	%
minimum		0,2	0,6	3,0
maximum		96,0	83,5	98,5

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Determination of Particle Size Distribution
QA / QC

Coefficient of variation for different concentrations - reference materials

Fraction	> 63 um		2 - 63 um		< 2 um	
	%	CV(%)	%	CV(%)	%	CV(%)
minimum	**2,16**	**6,17**	**10,50**	**10,62**	**5,20**	**19,60**
maximum	**95,40**	**0,92**	**82,37**	**0,29**	**58,98**	**5,33**

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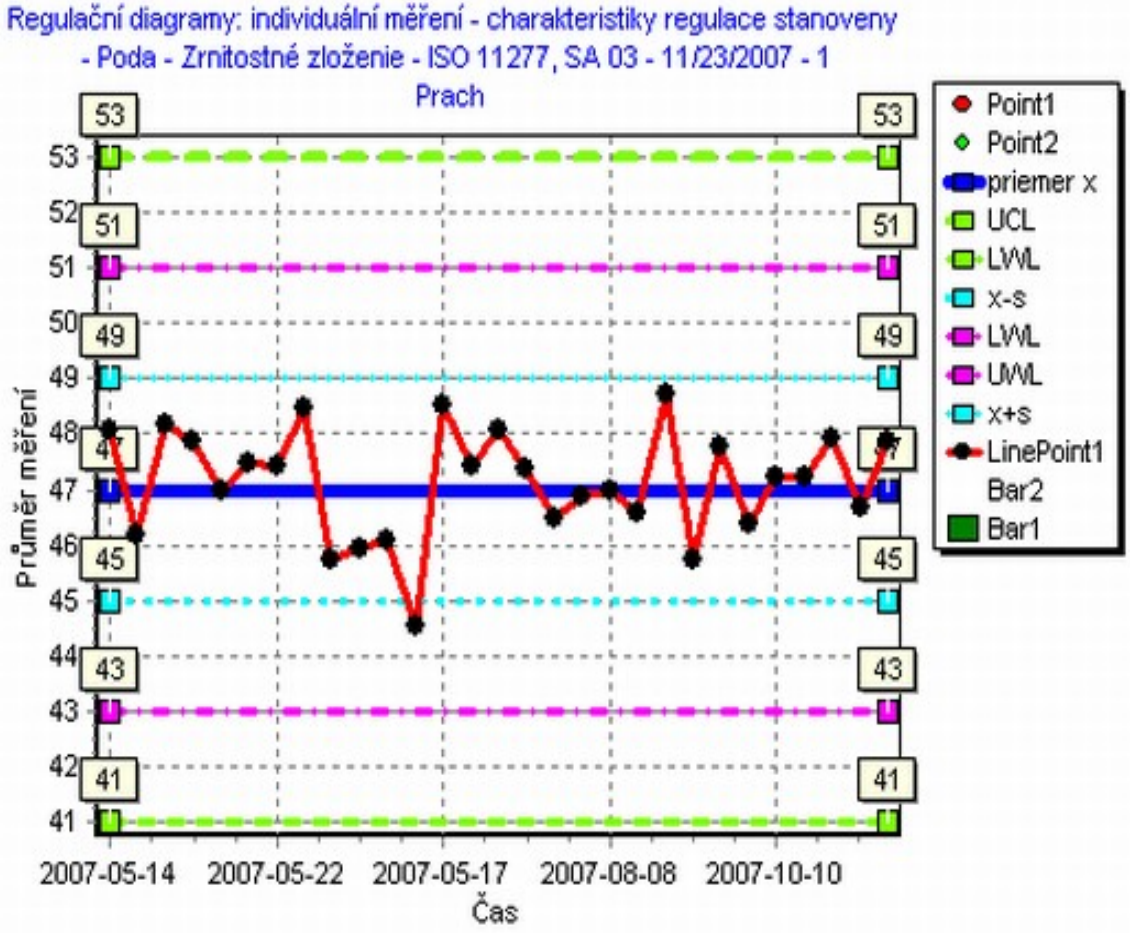
QA /QC



Charakteristika	Hodnota
Vypočtená hodnota	47,00
St. nejistota	0,89
Rel. st. nejistota (%)	1,89
Faktor pokrytí	1,96
Rozšířená st. nejistota	1,74
Rel. rozšířená nejistota	3,70

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Acknowledgements

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Thanks for attention.

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