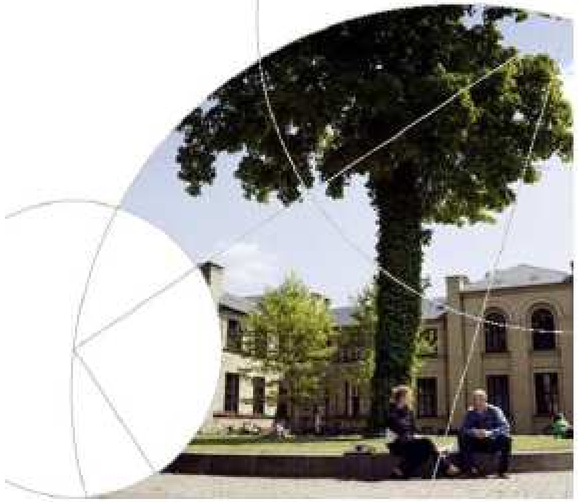
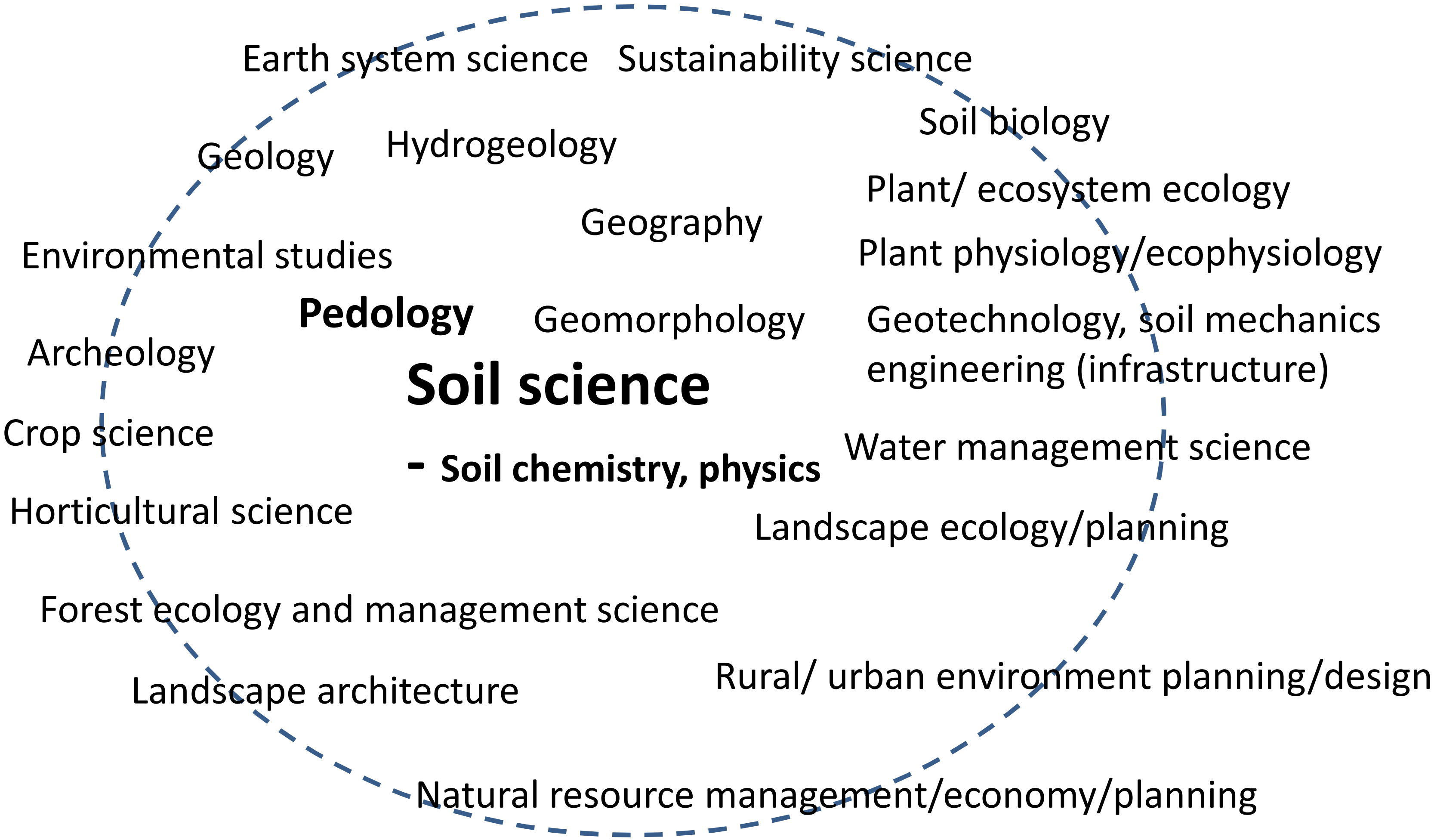
International DFJ seminar
Thursday 28. May 2015, 10-12 a.m. (GMT+1)
Soil texture and measurement of particle size distribution – state of the art and novel methods



Program

- 10.15 – 10.30 ‘Introduction and welcome.’ Vibeke Ernstsen.
- 10.30 – 10.45 ‘Soil texture: Some methods and problems.’ Ole K. Borggaard.
- 10.45 – 11.15 ’Measurement of soil PSD’s by hydrometer/sieving and laser diffraction using two different instruments’. Ingeborg Callesen, KU.
- 11.15 – 12.00 Discussion about R&D needs, and networking activities.
- 12.00 Lunch

Soil science today – all fields are interested in soil functions related with **soil texture**



Example from forest ecology

Ecosystems (2002) 5: 385–398
DOI: 10.1007/s10021-001-0082-4

ECOSYSTEMS

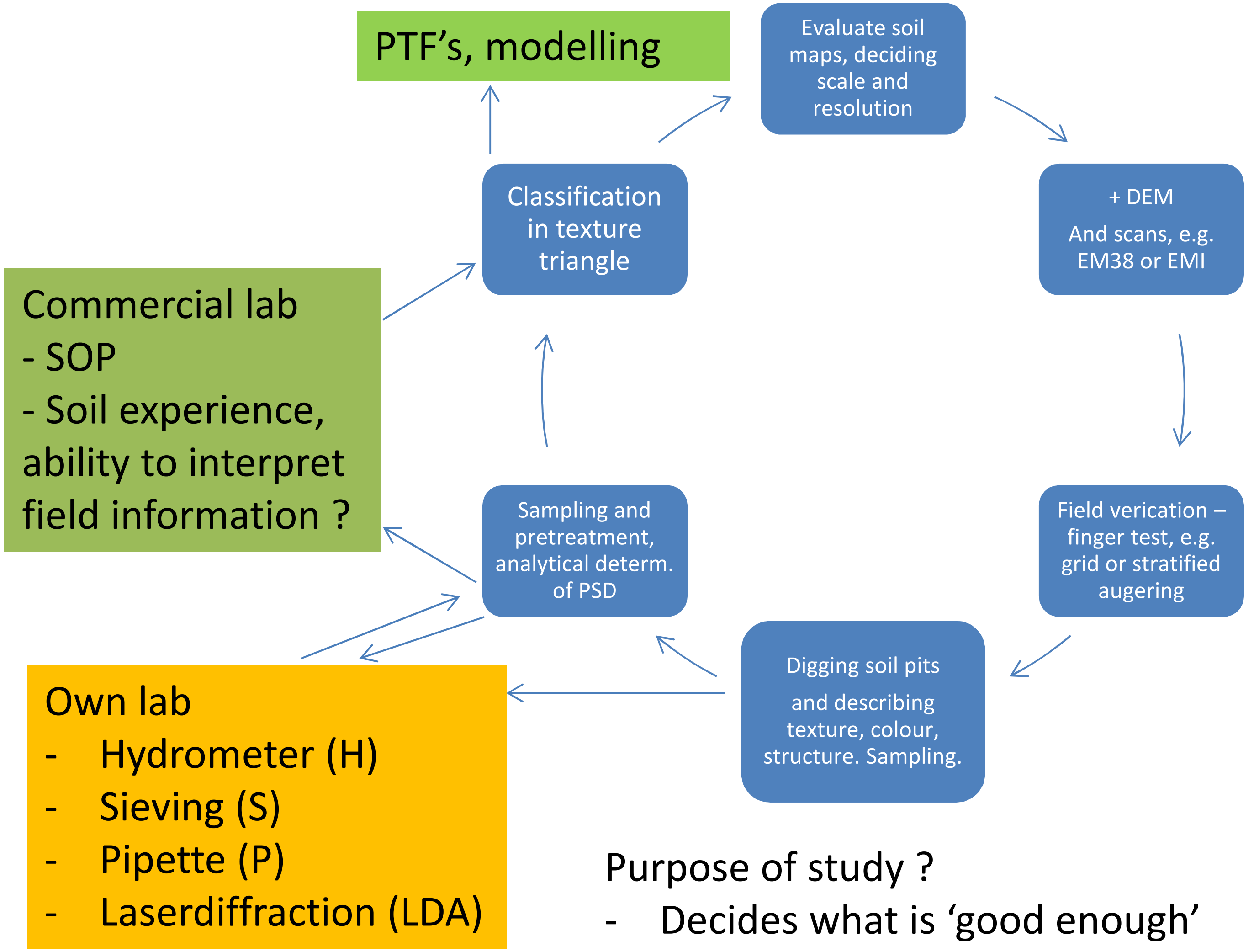
Tree Species Effects on Calcium Cycling: The Role of Calcium Uptake in Deep Soils

Feike A. Dijkstra[1,2]* and Mark M. Smits[1]

'' At Camp Pond, soil texture at three different soil depths, analyzed on a Coulter LS230 laser grain-sizer (Buurman and others 1997), was similar among sites (Table 1)''

That's all…

Background: Landscape management based on soil information - workflow



Purpose of study ?

- Decides what is 'good enough'
- Input to pedotransfer functions with model uncertainty

Laser diffraction as an alternative to hydrometer and sieving

2014: Internal project group at KU, Faculty of Science,

Department of Geosciences and Natural Resource Management (IGN)

- Ingeborg Callesen and Lars Vesterdal, Section for Forest, Nature and Biomass
- Thorbjørn Joest Andersen, Jesper Bartholdy and Henrik Breuning-Madsen, Section for Geography, IGN

Two instruments

- Malvern Mastersizer 2000 at Section for Geography, KU, cared for by Thorbjørn Joest Andersen and laboratory coordinator Vagn Moser.
- Sympatec Helos , Dep. Geosciences, Aarhus University, laboratory leader Charlotte Rasmussen, Bente Rasmussen and Søren Munck Kristiansen

Aim of study – Particle size distributions of sediments (texture)

- Is laser diffraction an alternative to hydrometer (H) and sieving (S) ?
- Is the equivalent diameter for clay of 8 my general?
- Is the 'specific surface area' provided by different instruments useful and comparable ?
- What operating procedures are used – pre-treatment actions and during analysis ?

Two laser diffraction instruments

- Malvern Mastersizer 2000, range 0.02 my to 2 mm, wet and dry unit

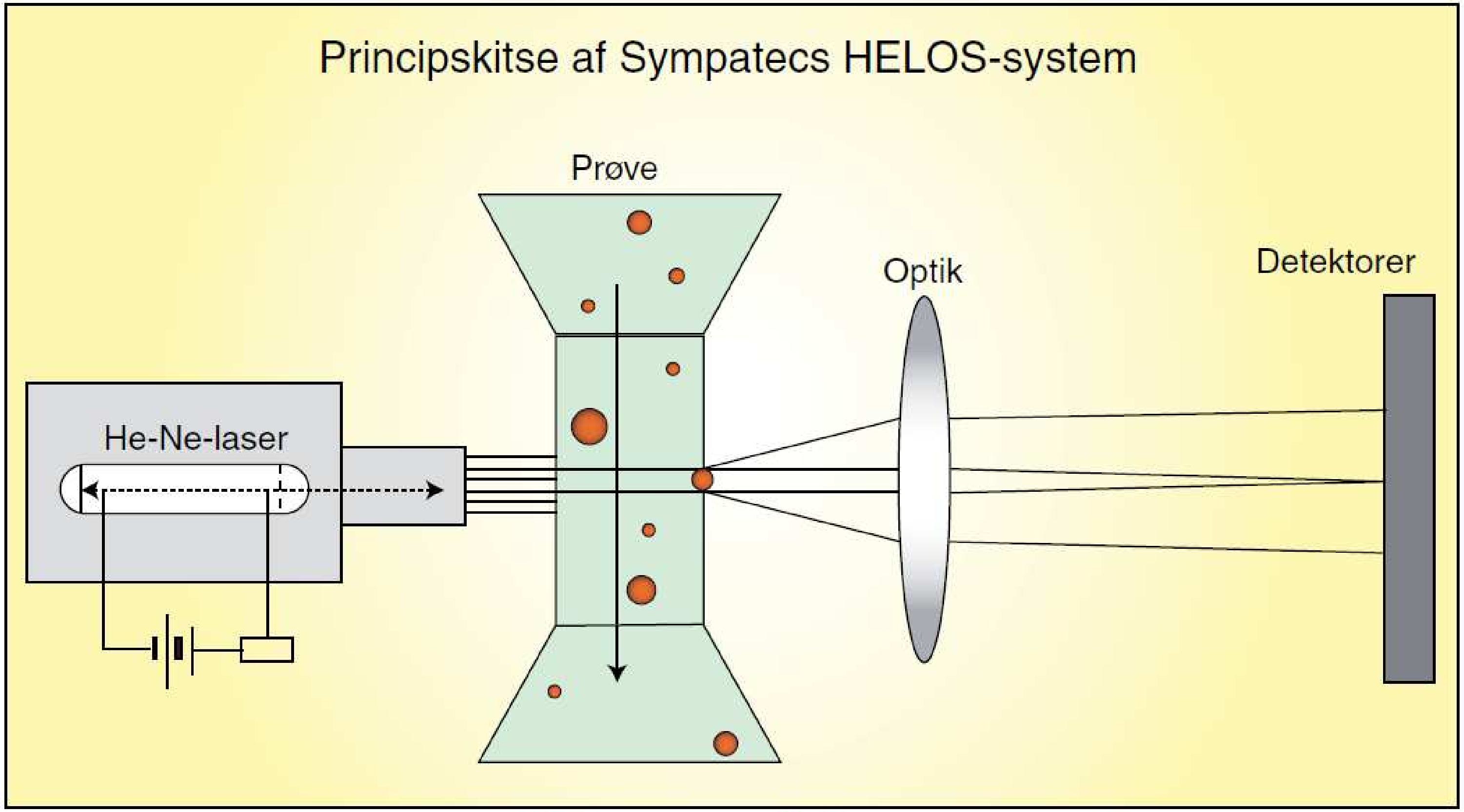
Mastersizer 2000 technical specifications

Optical Unit	Specification
Size range	Materials in the range 0.02µm to 2000µm
Measurement principle	Mie scattering
Detection systems	Red light: forward scattering, side scattering, back scattering Blue light: wide angle forward and back scattering
Light sources	Red light: helium-neon laser Blue light: solid-state light source
Optical alignment system	Automatic rapid align system with dark field optical reticle
Sample dispersion unit interchange	Sample dispersion units automatically recognized, configured and enabled on insertion of measurement cell cassettes into sizer
Laser system	Mastersizer 2000: Class 1 laser product Autosampler 2000: Class 2 laser product

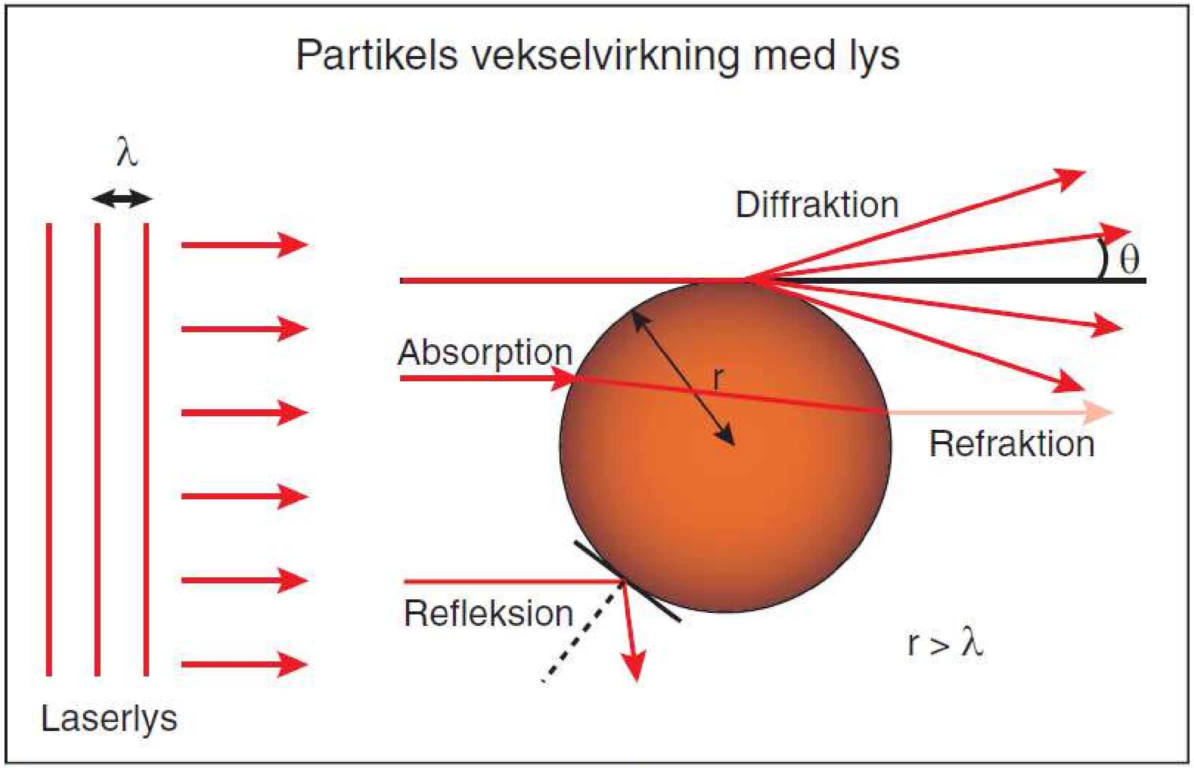
- Sympatec Helos, range 0.0018 mm – 3.5mm, three lenses
  - Wet unit
  - Gravimetric dry unit

Laserdiffraktometer fra Sympatec placeret ved Geologisk Institut i Aarhus. (Foto: Charlotte Rasmussen)

C. Rasmussen og K. Dalsgaard, Geologisk nyt 6/10



Simplificeret skitse af Sympatecs HELOS-system. He-Ne laseren udsender rødt lys med en bølgelængde på 632,8 nm. (Grafik: UVH modificeret efter Charlotte Rasmussen)



Fiktiv situation hvor lys med en given bølgelængde og med et parallelt strålebundt rammer en sfærisk partikel, der er optisk tættere end det omgivende medium. Radius (r) er i alle tilfælde meget større end bølgelængden (λ). (Grafik: UVH modificeret efter Charlotte Rasmussen og Sympatec)

Mie theory – best for <10 my

Forest soil samples
Data (hydrometer and sieving (% w)

					Clay	Silt	Fine sand	Coarse sand		Coarse sand
Lab_id	Lok2	Hor	cm	cm	0 - 2 my	2 - 20 my	20 - 200 my	200 - 1400 my		1400 - 2000 my
1875	Hastrup	Appl	0	-20	3	2	28	66.6		0.4
1882	Sønderborg	B(g)1	27	-68	38	37	18	6.7		0.3
1889	Tranum	Bsg	5	-35	2	1	58	38.9		0.1
1892	Skjoldenæs-holm	E(g)	30	-53	10	13	38	34.0		4.0
1900	Bregentved	Btg	110	-120	22	18	39	20.1		0.9
2042	Valløø	Bt(g)2	68	-110	15	11	41	30.3		1.7
2047	Kragelund	Bs	40	-60	7	7	38	46.4		1.6
2059	Viemose	E	11	-43	18	20	40	19.4		2.6

Test: Marine sediments

ID	Description
D3	Sea floor sediment from Disko Fjord, W Grønland. Low in OM
D4	Sea floor sediment from Disko Fjord, W Grønland. Very low in OM
G23	Sea floor sediment from Chile
MF13/1	Sea floor sediment from Mariager Fjord, high in OM
MF1201	Sea floor sediment from Mariager Fjord, high in OM
NP77	Sea floor sediment from Nha Phu bay, SE Vietnam
S5	Sea floor sediment from Sermilik fjord, SE Grønland. Very low in OM.
S10	Sea floor sediment from Sermilik fjord, SE Grønland. Very low in OM.
S11	Sea floor sediment from Sermilik fjord, SE Grønland. Very low in OM.
SK12	Sediment from core in marsh. High in OM
SK13	Sediment from core in marsh. High in OM

OM ~ organic matter

Malvern Mastersizer 2000 one lens, 72 size fractions (bins)

Operating procedure in Sektion for geografi, described by Vagn Moser
Analysis using wet unit, Hydro S, Mie theory

- If the sample contains more than 5% carbon it is removed with H_2O_2 (time consuming). Check for carbonates by HCl and salts by EC (SOP)
- After end-over turning of the soil container, sample ¼ spoon soil (0.3 – 0.5 g) depending on clay and silt content – visual and sensoric inspection.
- The sample is wet-sieved (using 0.1 M $Na_4P_2O_7$) through a 1.4 mm sieve into a 250 ml beaker, since larger particles may get stuck in the flow cell.
- The beaker is subject to 2 min ultrasonic treatment, full power.
- The dispersion is transferred to the Malvern 2000 Hydro S unit.
- The sample is measured in five runs, and three are picked for averaging
- Results are treated in a spreadsheet template
- *If relevant the soil is dry sieved on a 1.4 mm sieve, after additional gentle grinding, and laser results are adjusted accordingly by recalculation.*

Further details on instrument set-up. Thorbjørn and Vagn.

Sympatec Helos

Same pretreatment measures as at KU, but up to you.

Sediments are separated in 3 fractions:
(1) < 0.063 mm – wet sieving after dispersion in 0.1 M $Na_4P_2O_7$ and overnight end-over shaking.
(2) 0.063 - 0.250 mm after drying, separated from (3) 0.250 - 2 mm by sieving on a 250 my sieve.

50 g sample used -> One 500 ml bottle and two bags with coarse silt/sand with known weight.

(1) Is subsampled with a ca. 5-10 ml plastic tub after 2 min ultrasonic treatment. Wet sampler unit, lens R4 – range 0,0018-0,350mm – 31bins
(2) Mixed with water to a paste, and a spatel size sample is transferred to the wet unit , lens R7 – range 0,018-3,5mm – 31 bins
(3) Measured in full quantum via the gravimetric unit on a lens R7, 31 bins.

A particle size distribution is calculated based on the three fractions and their masses by the lab, choosing the 'best' (unflawed) of three runs for each fraction.

Further details on set-up : Charlotte Rasmussen, Bente Rasmussen, Søren M. Kristiansen, Dep. Geosciences , AU.

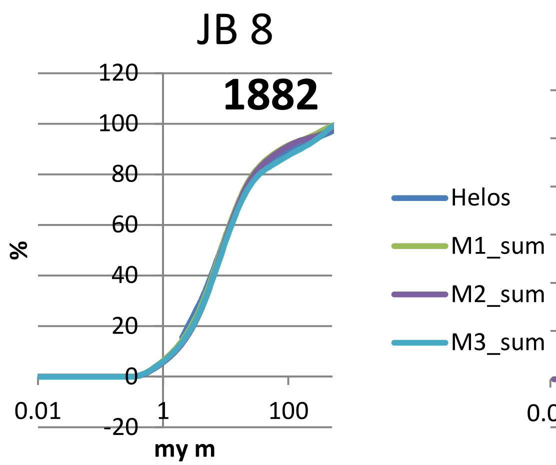
Results – forest soils comparison of H/S and LDA w. 8 my clay eq.

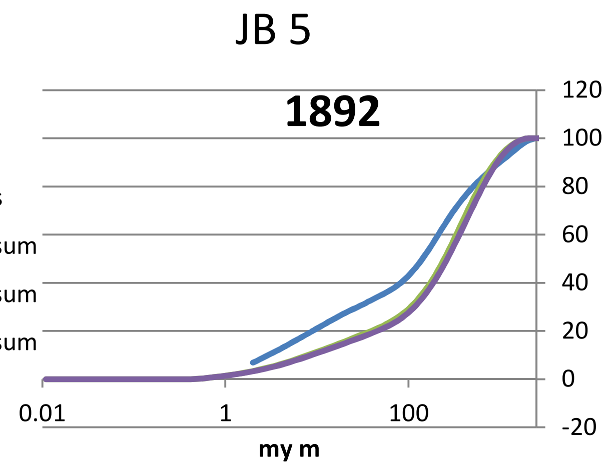
JB	Prøve	Ler <2 (8) µm	Std	Silt 2(8)-20 µm	Std	FS 20-200 µm	Std	GS 200-1400 µm	Std	Sand >20 µm	Std
1882	IGN, Mastersizer	43.3	1.0	27.2	0.4	22.2	0.4	7.3	1.8	29.5	1.4
	AU, Helos	49.0		24.9		18.6		7.5		26.1	
8 - Svær lerjord	Hydr/Sigte	38.0		37.0		18.0		7.0		25.0	
1892	IGN, Mastersizer	9.8	0.3	5.6	0.2	27.4	0.7	57.2	1.1	84.6	0.5
	AU, Helos	19.0		8.4		30.1		42.6		72.7	
5 - Grov sandbl. Lerjord	Hydr/Sigte	10.0		13.0		38.0		38.0		76.0	
1900	IGN, Mastersizer	19.4	1.8	10.2	1.1	34.7	2.6	35.7	5.4	70.4	2.9
	AU, Helos	29.6		12.4		32.6		25.5		58.1	
7 - Lerjord	Hydr/Sigte	22.0		18.0		39.0		21.0		60.0	
2042	IGN, Mastersizer	14.3	3.3	8.8	2.3	31.2	2.1	45.6	7.4	76.9	5.6
	AU, Helos	18.2		8.9		32.8		40.1		72.9	
7 - Lerjord	Hydr/Sigte	15.0		11.0		41.0		32.0		73.0	
2047	IGN, Mastersizer	4.6	0.6	2.3	0.2	22.3	1.9	70.7	1.8	93.1	0.8
	AU, Helos	8.8		4.3		31.7		55.2		86.9	
3 - Grov lerbl. sandjord	Hydr/Sigte	7.0		7.0		38.0		48.0		86.0	

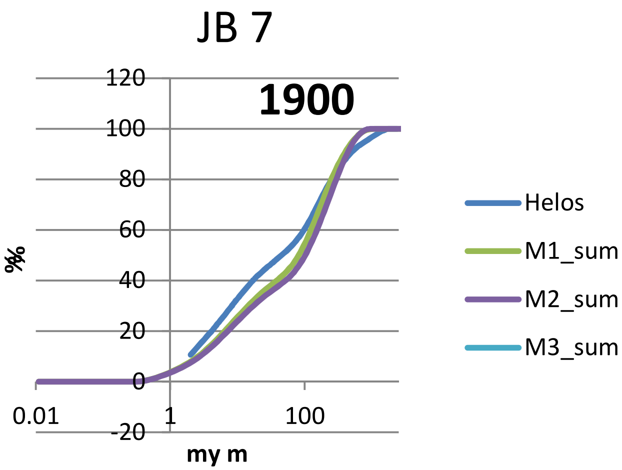
‘Method’ repeatability

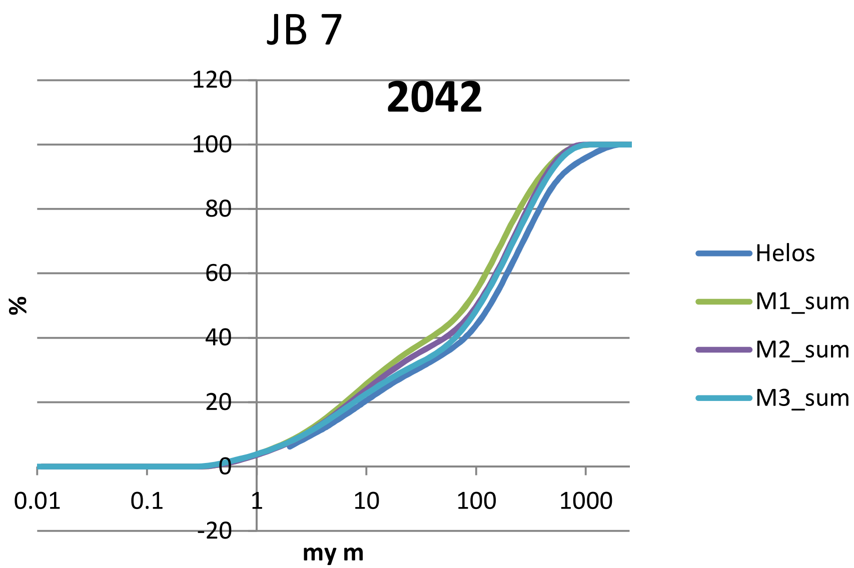
* Malvern Std is based on three repeated samplings from container

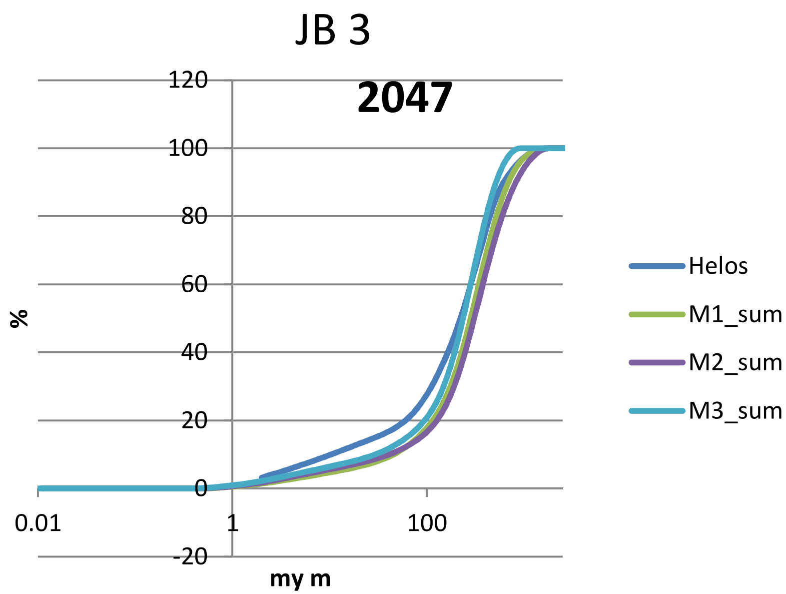
Results – forest soil samples







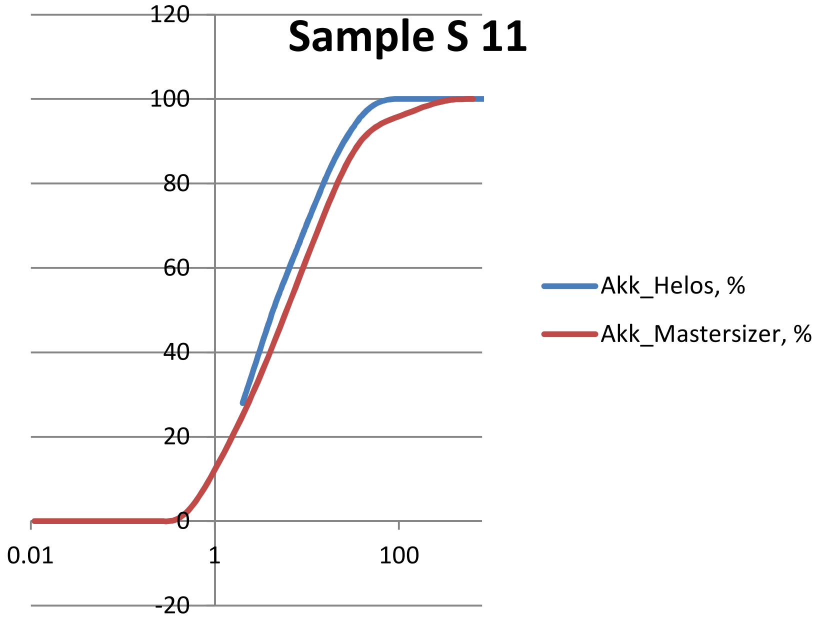


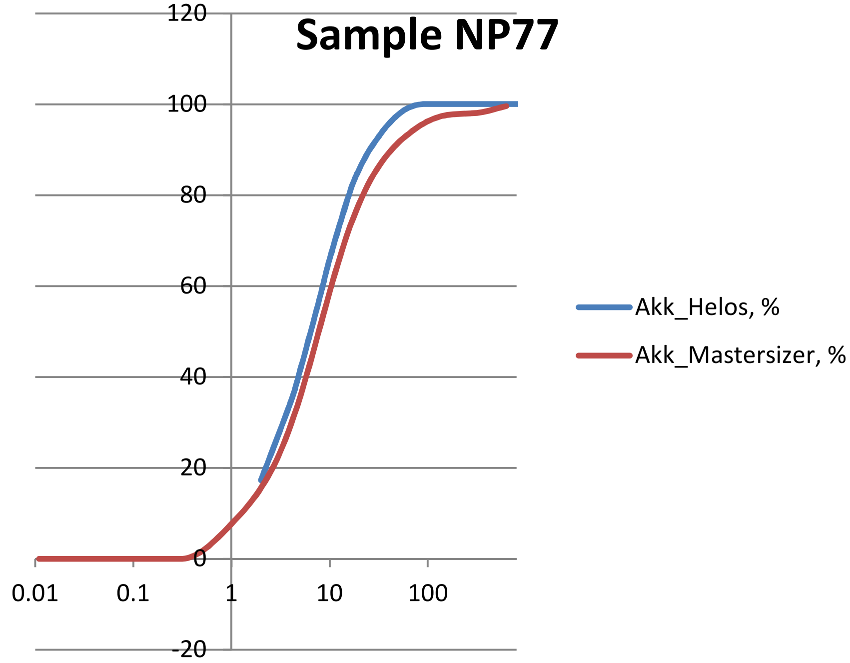


Method repeatability M1-M3 – instrument repeatability not shown (average of three)

Reproducibility: Helos vs M1 – M3

Results – Marine sediment PSD's

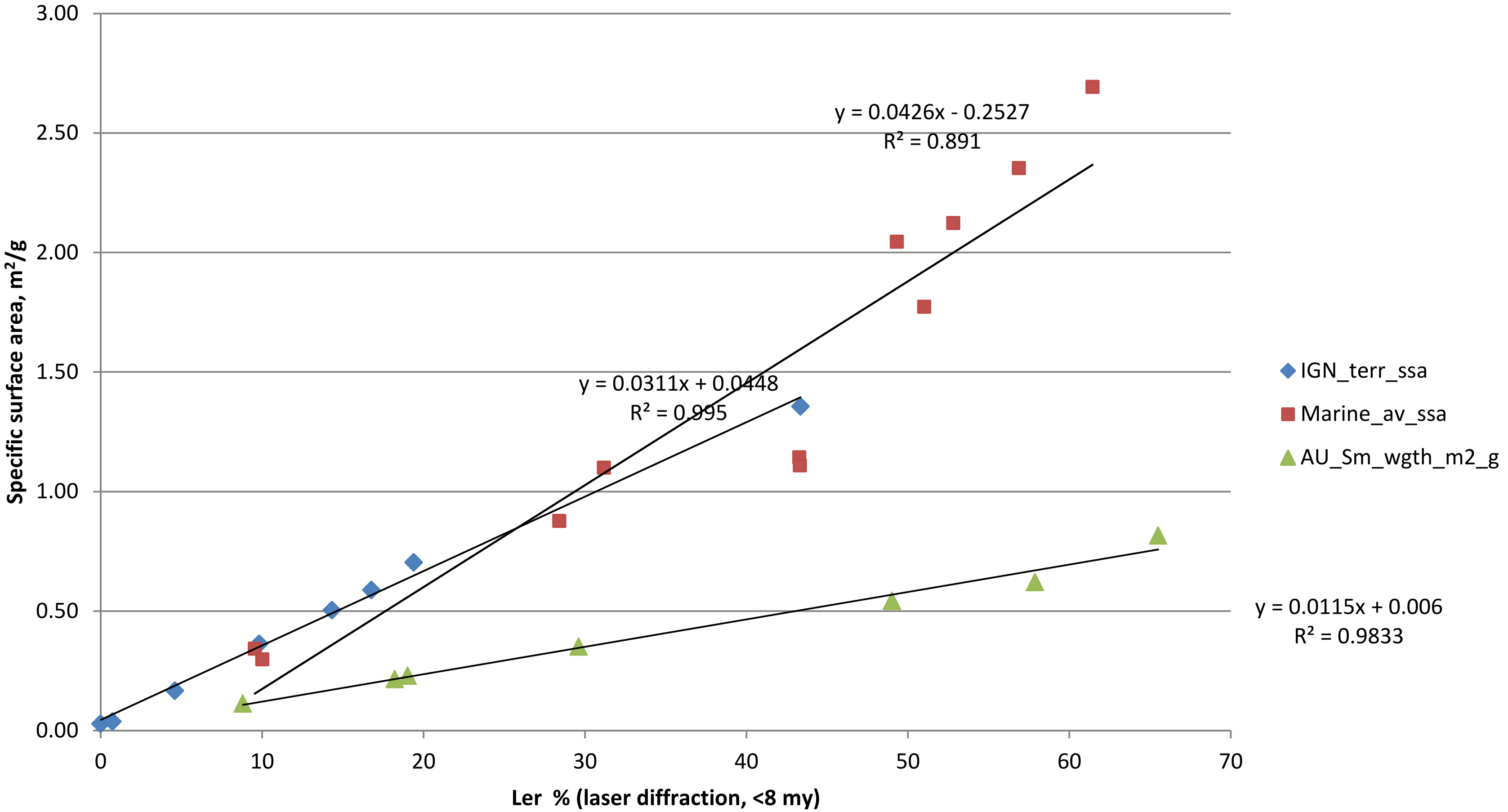




MS – avg. of 3 best out of 5

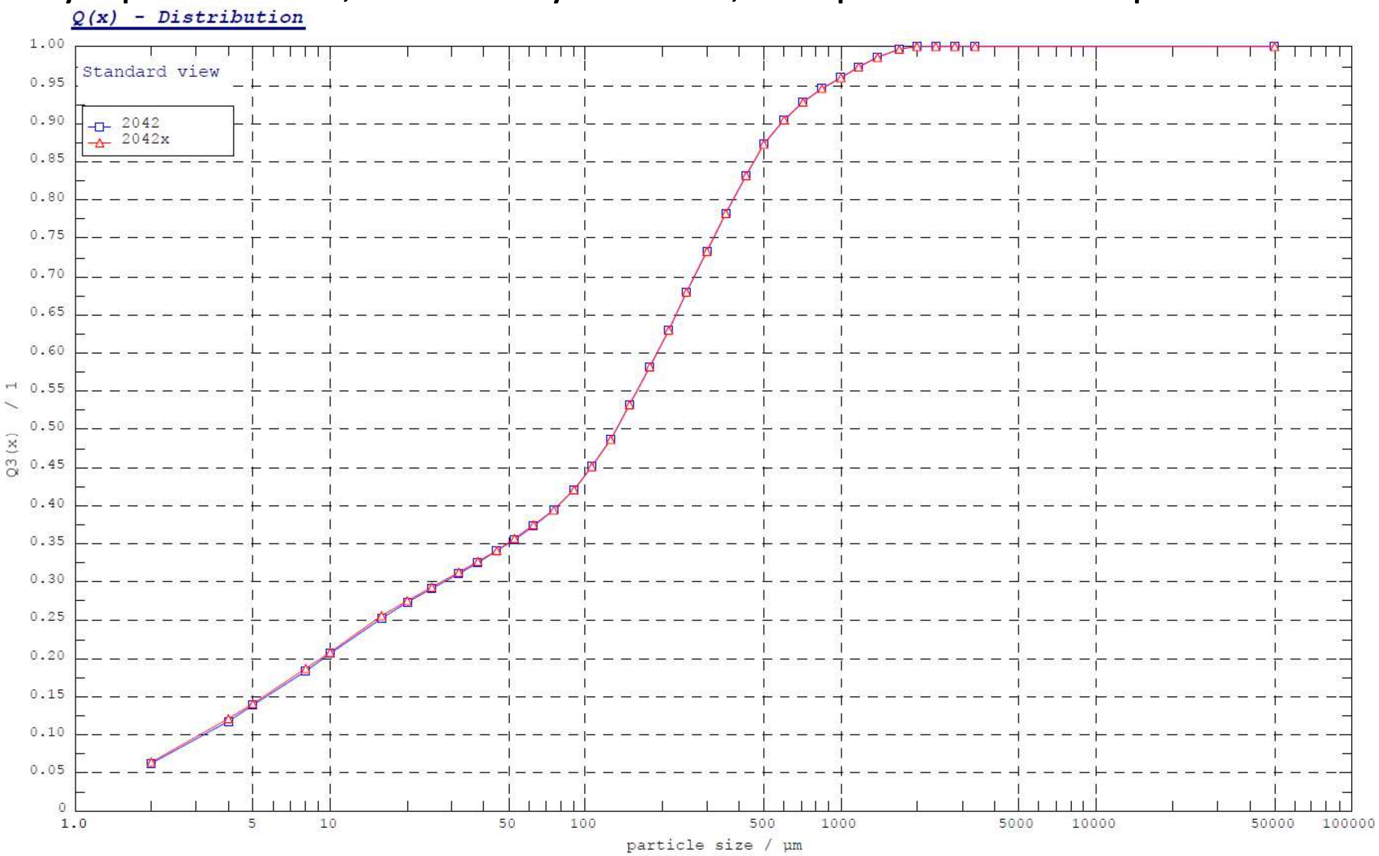
Helos – best of 3 – e.g. avoiding signs of air bubbles

Results – 'specific surface area'



Sampling uncertainty - Helos

Sympatec helos, <0.063 my fraction, sample 2042 - sampled twice

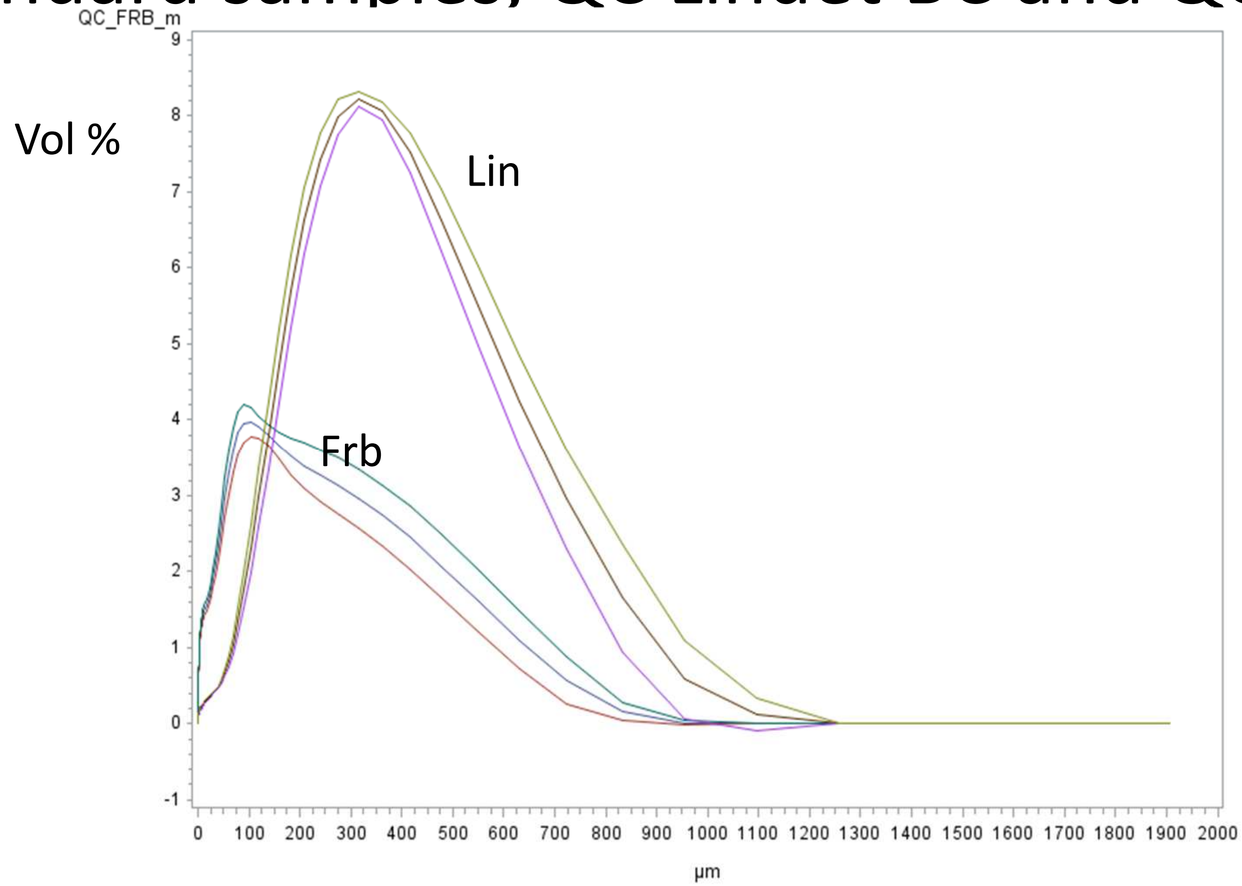


Method repeatable on the instrument – reproducible on other instruments

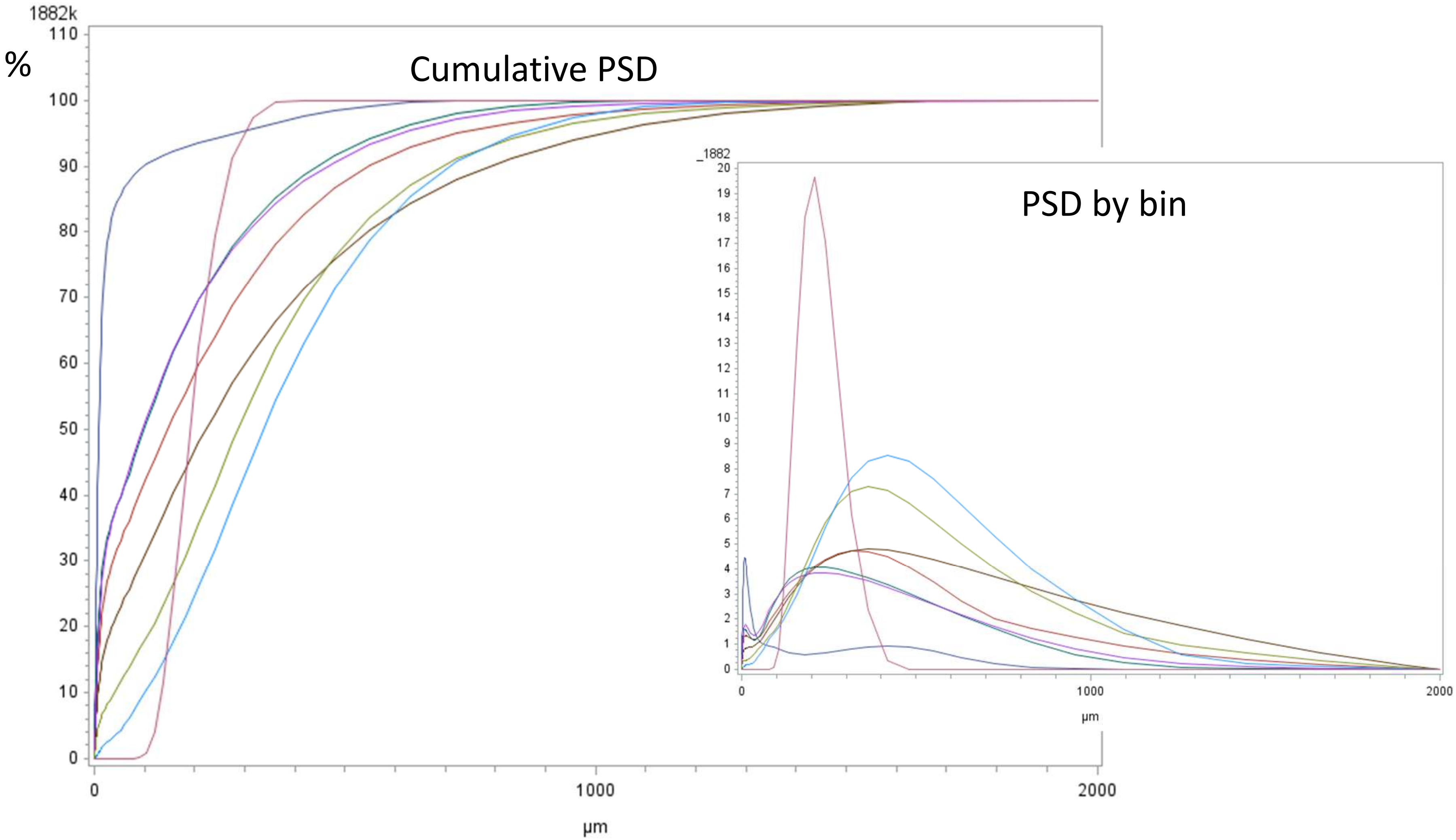
Sampling uncertainty Malvern

- Two standard samples, QC Lindet BC and QC Frb A

Mean +/- 1 SD, N=3



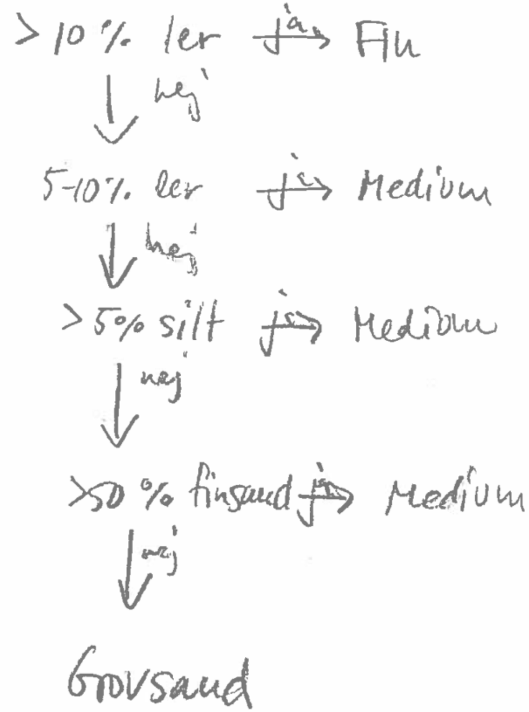
Why sieve based bins and texture triangles, when we can have PSD's ??



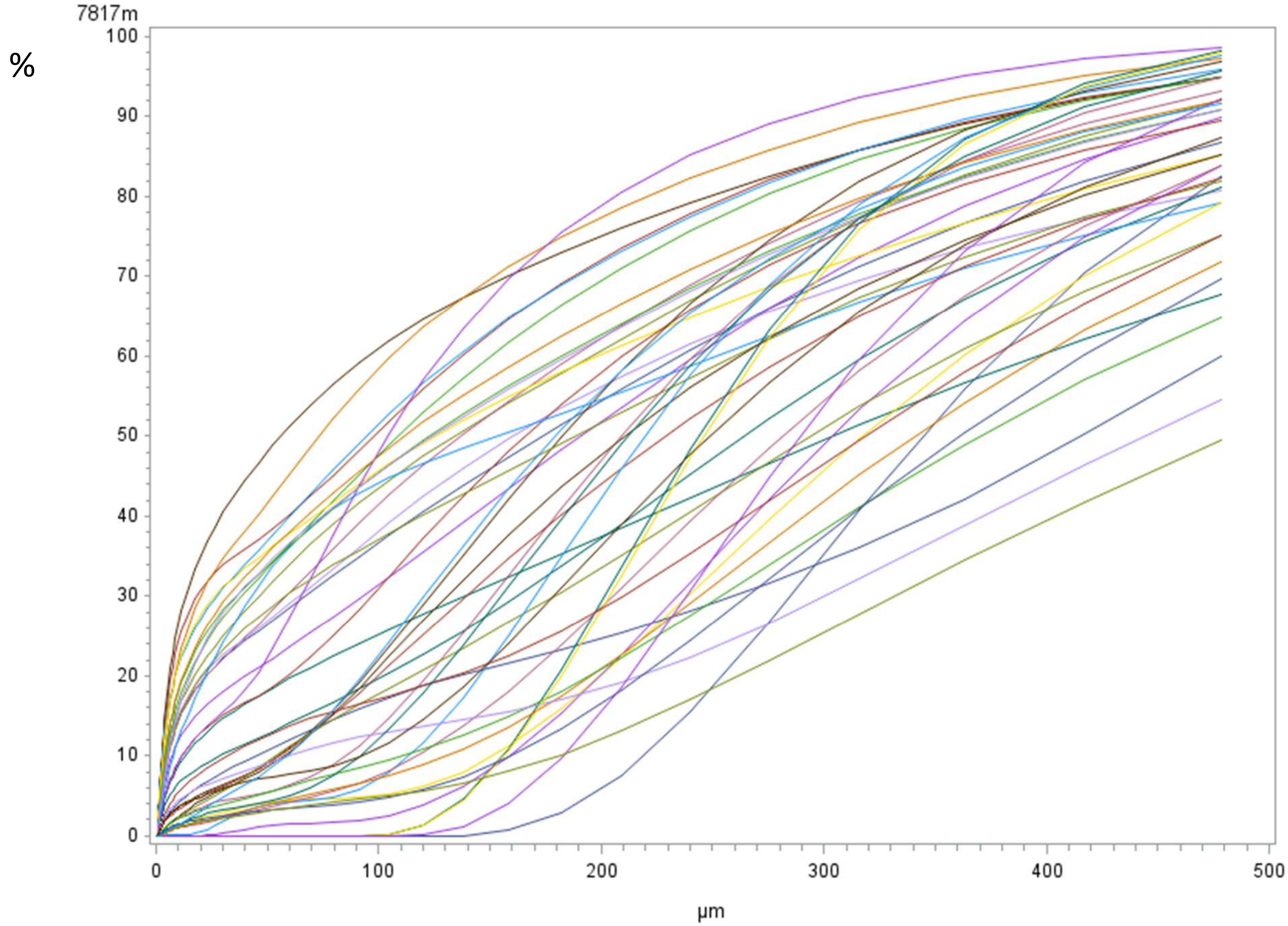
Test of texture class assessment by finger test and laser diffraction method and : 50 samples from National Forest inventory

Forest

FOREST	Jordtype	Teksturdefinition for jordtype	Symbol	JB-nr.	Vægtprocent Ler under 2 µm	Silt 2-20 µm	Finsand 20-200 µm	Sand, ialt 20-2000 µm	
COARSE	1	Grovsandet jord	GR.S.	1	0-5	0-20	0-50	75-100	
MEDIUM	2	Finsandet jord	F.S.	2			50-100		
	3	Grov lerblandet sandjord	GR.L.S.	3	5-10	0-25	0-40	65-95	
		Fin lerblandet sandjord	F.L.S.	4			40-95		
FINE	4	Grov sandblandet lerjord	GR.S.L	5	10-15	0-30	0-40	55-90	
		Fin sandblandet lerjord	F.S.L.	6			40-90		
Perhaps w.	5	Lerjord	L	7	15-25	0-35		40-85	
$CaCO_3$	6	Svær lerjord	SV.L.	8	25-45	0-45		10-75	
		Meget svær lerjord	M.SV.L.	9	45-100	0-50		0-55	
		Siltjord	SI.	10	0-50	20-100		0-80	
Humus	7	Humus	HU.	11					Over 10
	8	Speciel jordtype	SPEC.	12					



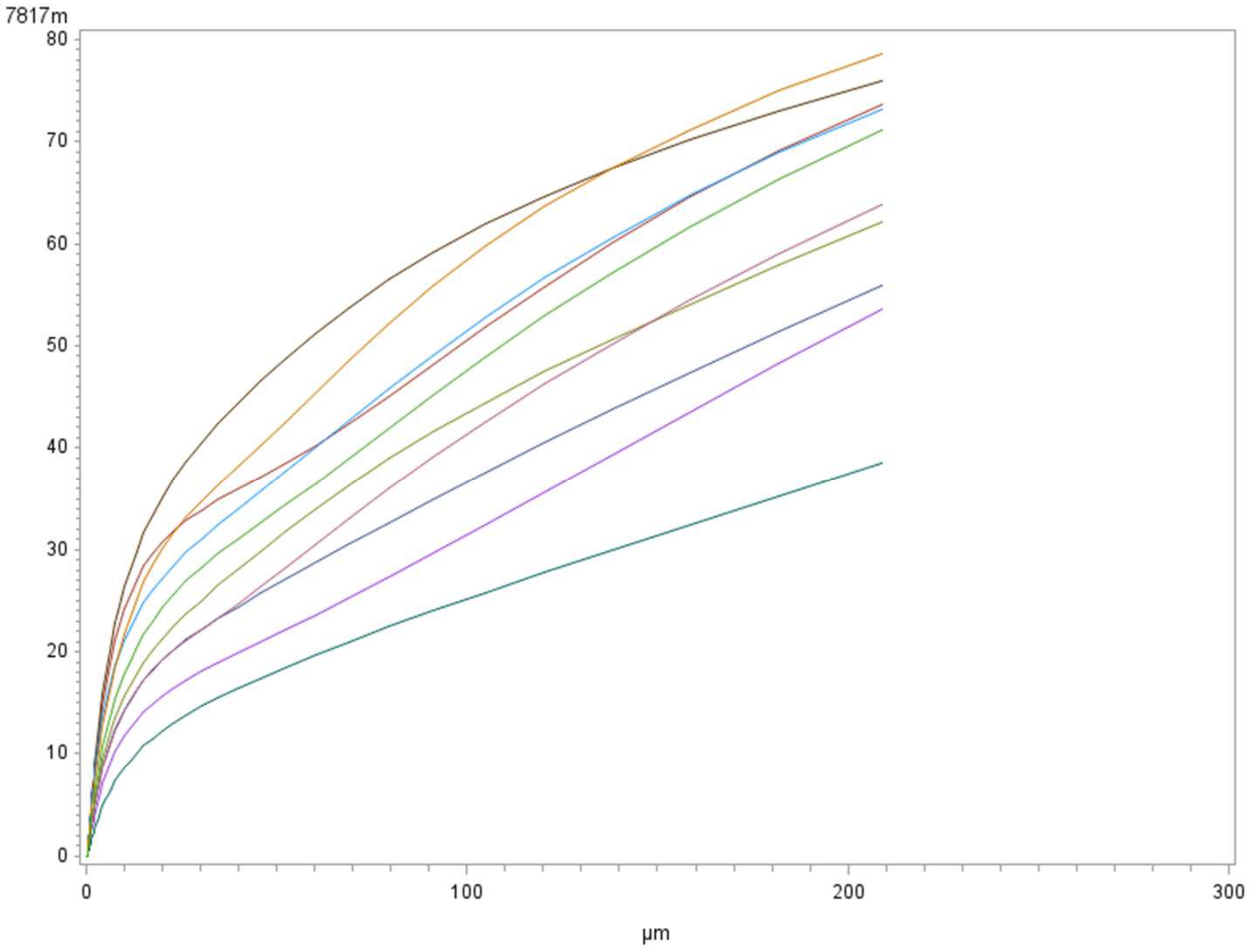
Clay < 2 my, silt 2-20 my, fine sand 20-200 my, coarse sand 200-2000 my



%
7817m
100
90
80
70
60
50
40
30
20
10
0
0
100
200
300
400
500
µm

How well can we classify ?
‘Fine, >10 % clay ‘

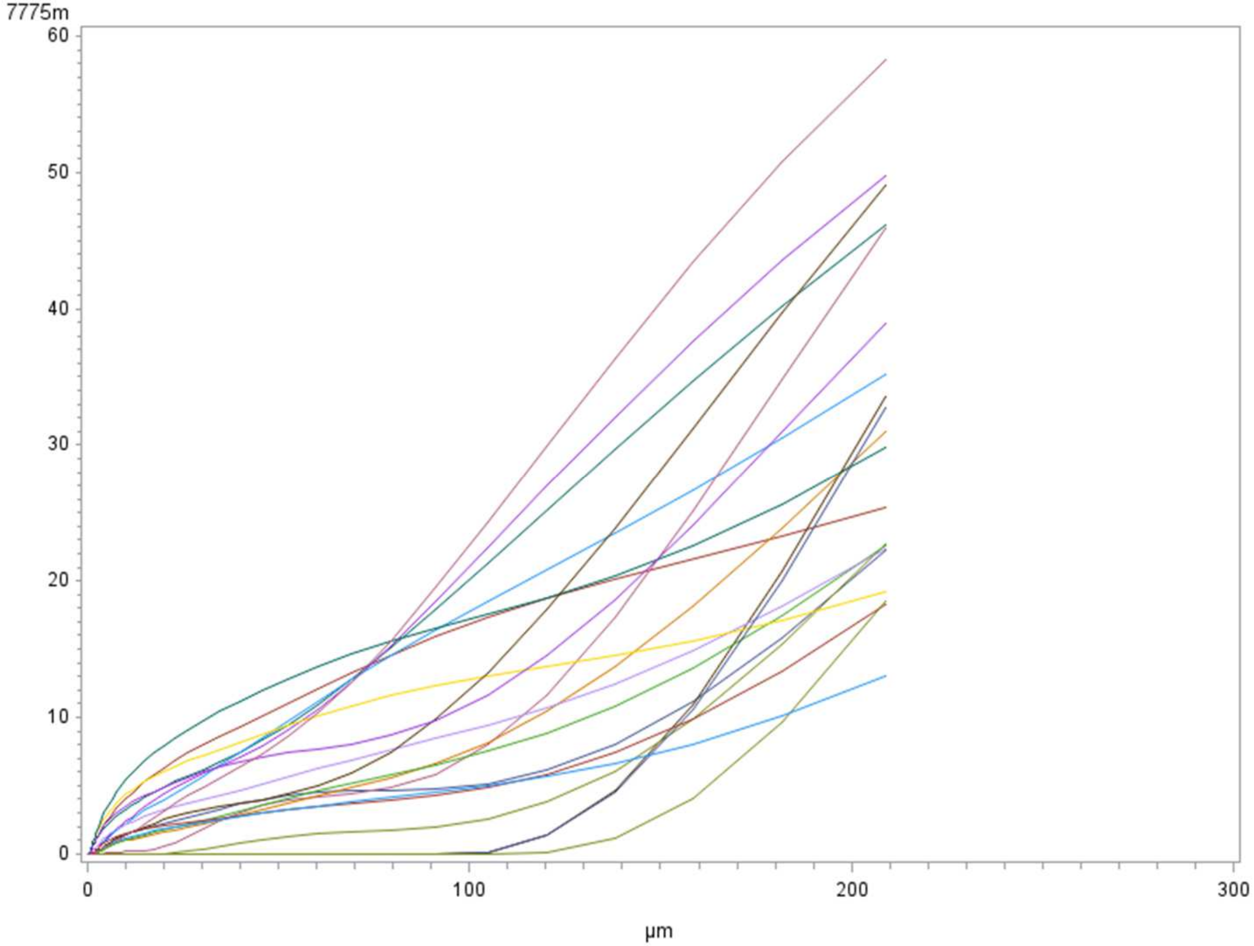
%



How well can we classify ?
‘Coarse ‘ :

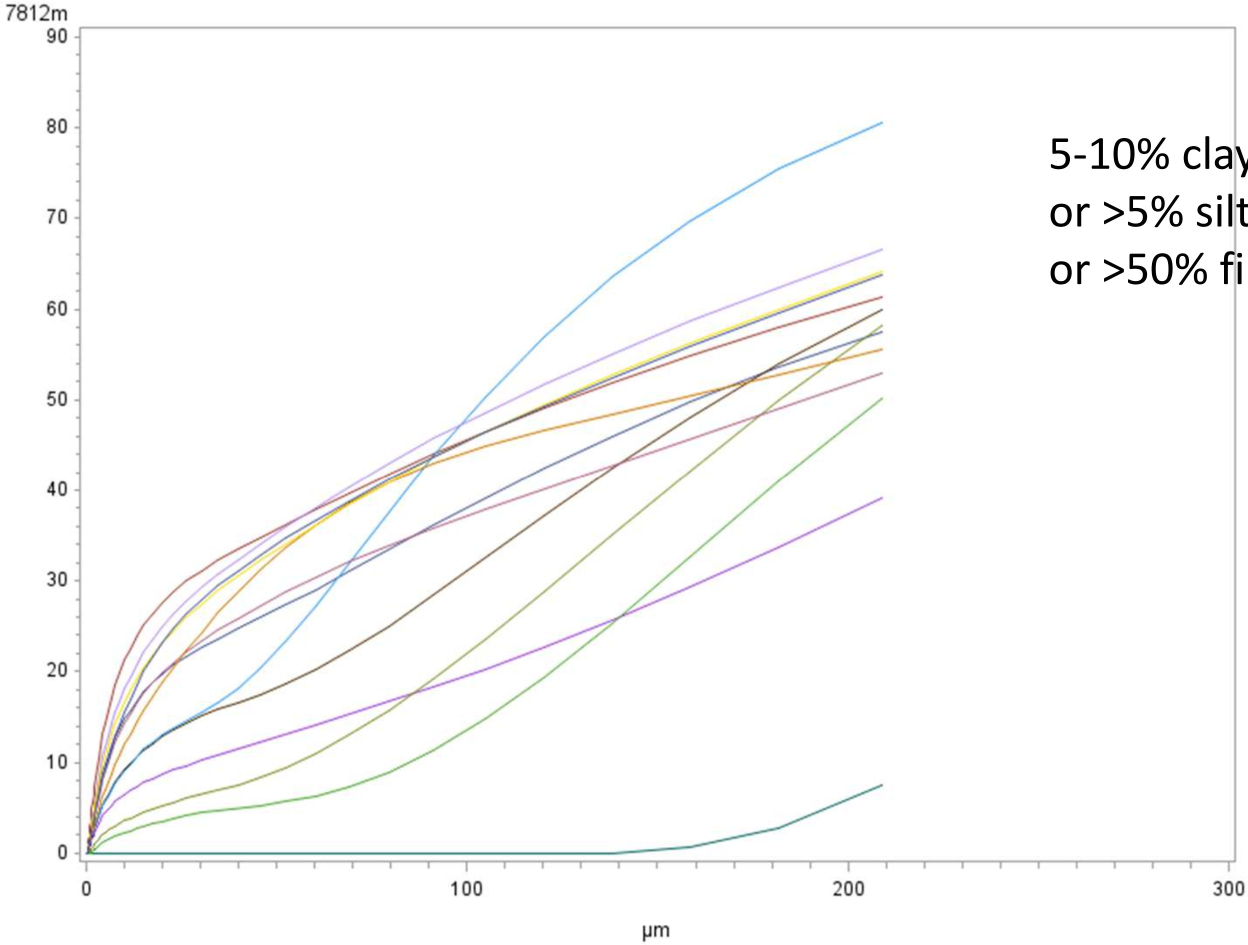
<5% clay, < 5% silt,>50% fine sand

%

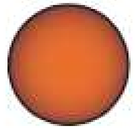


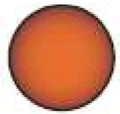
How well can we classify ?
'Medium '

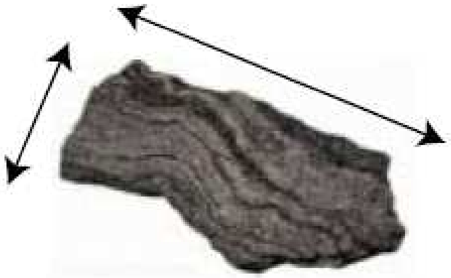
%



5-10% clay
or >5% silt
or >50% fine sand

Sfære der kan passere samme sigte-apertur:  d_{sieve}

Sfære med samme sedimentationshastighed:  d_{sed}

Ikke-sfærisk partikel: 

Rasmussen C. and Dalsgaard K, 2011

Literature

- DS/ISO 13320 – describes analytical procedures. Commercial.
- Konert and Vandenberghe, Sedimentology (1997). Suggest 8 my clay limit.
- Di Stefano et al Biosyst. Eng. (2009) – Demonstrates effects of H_2O_2 pretreatment and ultrasound, choice of Mie/Fraunhofer theory. Uses transfer function:

  Clay vol% (HS)=1.9 * Clay vol% (LDA)
- Rise og Brendryen, NGU (2013) - Compare sedigraph and LS Coulter 200 on marine and terrestrial sediments. Suggest equivalent clay content (4* <2my + <7.4my)/2
- Miller and Schaetzl, SSSAJ (2011) – examines repeatability of resampling – recommend using the two best runs and several bins.
- Pieri et al. , Geod. (2006). Bimodal gaussian PSD's. LDA understimates clay and overestimates silt.
- Taubner et al., J.Pl.N.So.Sci (2009). Transfer functions for H/S and LDA can be made, but misclassifications occur. Do not recommend LDA as a general method.
- Bah et al., SSSAJ (2009). Fitting performance for PSD's by laser, best overall model for cumulative PSD F(d) = a ln d + b.

Lab technician student work:

- Anne Kristin Sallerup: Kornstørrelsesbestemmelse – en sammenligning af to metoder, IGN, KU, 2014.
- More reports from students ?

More refs’

- Rasmussen, C. & Dalsgaard, Geologisk nyt (2011) Laserdiffraktion – bestemmelse af partikelstørrelsesfordeling

- Borggaard et al. Teksturanalyse – metode og udfordringer: In: Rubæk et al. Jordanalyser – kvalitet og anvendelse. DCA report nr 2, 2011.

Answer to ? PSD's of soil and marine sediments (texture)

- Is laser diffraction an alternative to hydrometer (H) and sieving (S) ?
  - Yes, but establish a set of reference PSD's for different sediment types
- Is the equivalent diameter for clay of 8 my general?
  - No, it seems to be depending on instrument, but is it important ?
- Is the 'specific surface area' provided by different instruments useful and comparable ?
  - Correlates with clay content, apparently not comparable between instruments. Does not add any new information.
- What operating procedures are used – pre-treatment actions and during analysis ?
  - Up to the soil sampling expert and lab technician in cooperation to choose pretreatment– preferably research labs with direct communication between technicians and soil scientists.
  - Soil committée in 2012 recommended H_2O_2 as standard pretreatment.
  - The Malvern OP should address the uncertainty introduced by small sample size and the possible misrepresentation of large particles 1000-2000 my.

Conclusion

- Instrument and method repeatability is good on both instruments
- Laser diffraction is fast, but size fraction separation is still time consuming (Helos) – no time gain in comparison with HS
- Fast analysis on Malvern may be ‘good enough’
- Results may not relate to absolute standards – but better method descriptions in literature is required to allow reproduction.
- Careful pretreatment procedures also re: ultrasound
- ‘specific surface area’ seem to be instrument specific
- Use PSD’s rather than method defined clay/silt/sand fractions
- New transfer functions for visual inspection and finger test to produce parameters for PSD’s?

A new network ☺

- Let’s work together on this in the future with a goal to establish new reference particle size distributions
- Explore further the use of LDA in e.g. agronomical, forestry and ecological studies