Lessons learned, and comparative measurements with the PARIO system for soil particle-size distribution

by
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consultation and comments by Wolfgang Durner are truly appreciated

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At a glance:

- How sensitive is the sedimentation-based PARIO system to vibration?
- How about changes in temperature?
- How repeatable are its measurements?

(key findings are on slide 8)
(not to miss: slides 3,4,6)

- How do its results compare to particle-size measurements by the pipette method and laser diffractometry?

(key findings are on slide 22)
(not to miss: slides 13,15,16,17,21)

The PARIO particle-size measurement system

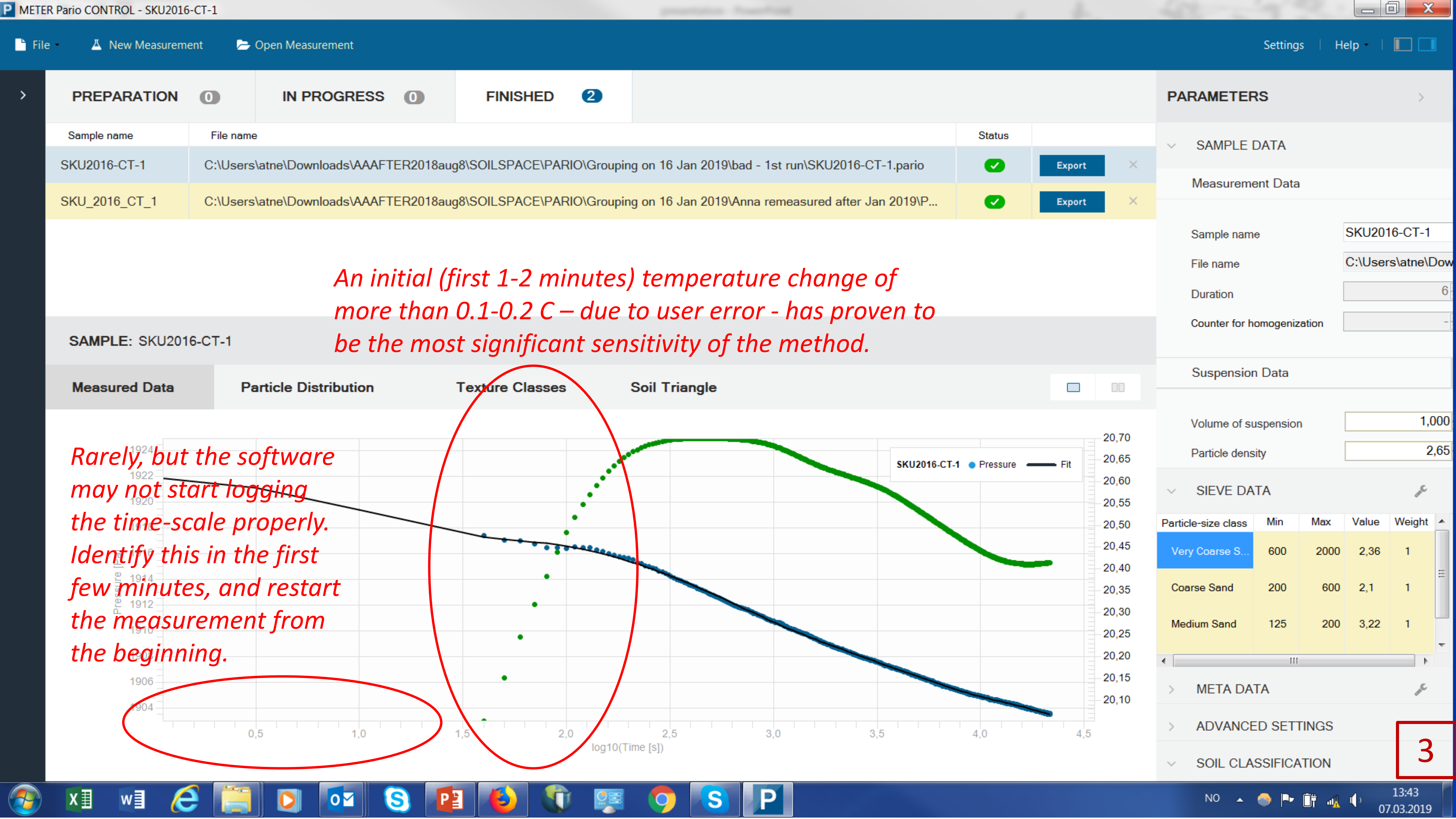
The change in pressure in the sedimenting solution is continually recorded and translated into a size-distribution of the sedimenting particles, based on Stokes' Law.

- Sample preparation should be the same as your 'standard' for pipette or hydrometer measurements

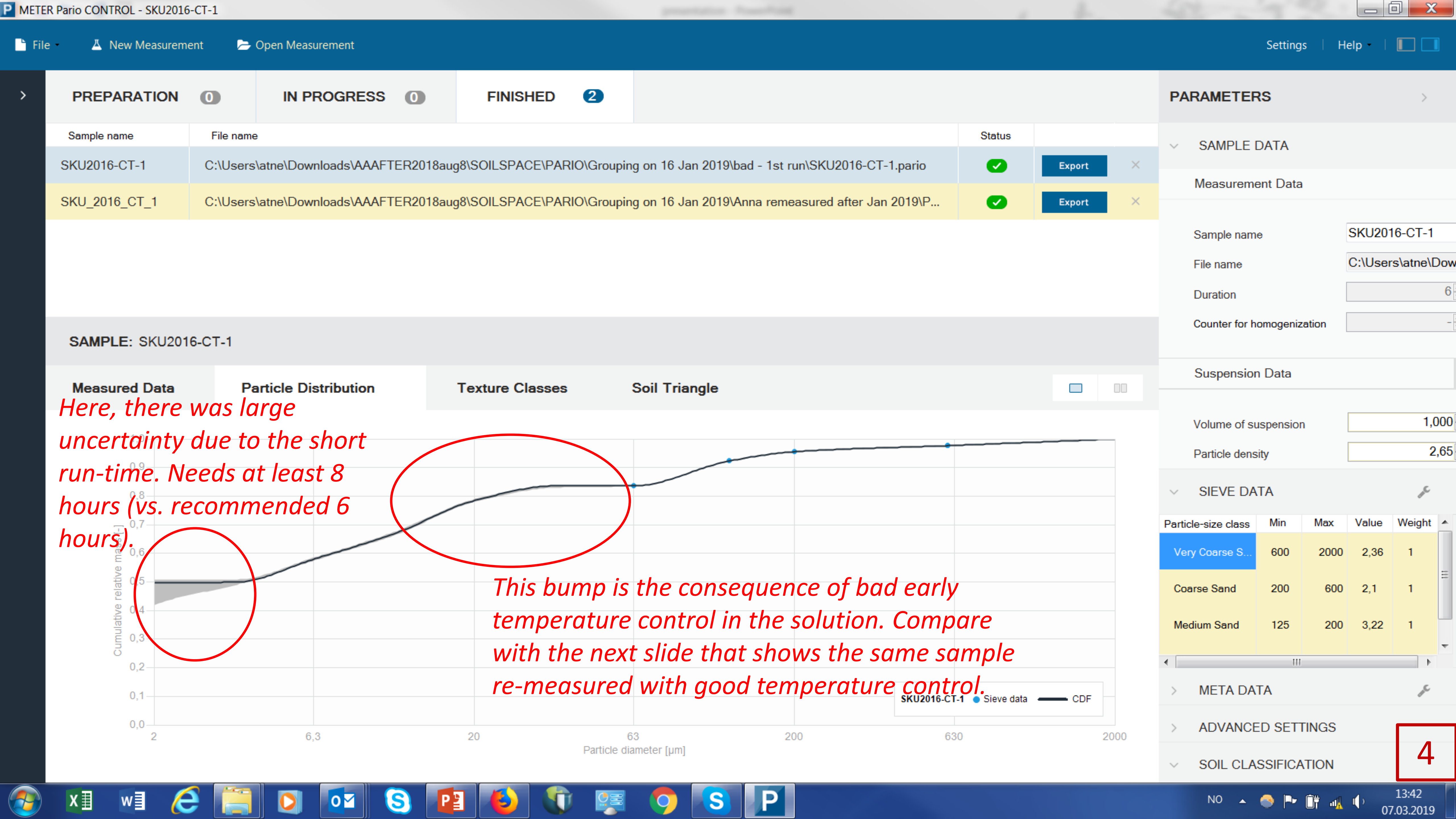
- Pressure sensor is moved into the shaken 1L solution in a timed fashion

- Sensors monitor pressure and temperature in the solution continuously for the <63 micron fraction

- Wet sieving is done the same way as for the standard measurements



METER Pario CONTROL - SKU2016-CT-1
File
New Measurement
Open Measurement
Settings
Help
PREPARATION 0
IN PROGRESS 0
FINISHED 2
PARAMETERS
Sample name
File name
Status
SKU2016-CT-1
C:\Users\atne\Downloads\AAAFTER2018aug8\SOILSPACE\PARIO\Grouping on 16 Jan 2019\bad - 1st run\SKU2016-CT-1.pario
Export
SKU_2016_CT_1
C:\Users\atne\Downloads\AAAFTER2018aug8\SOILSPACE\PARIO\Grouping on 16 Jan 2019\Anna remeasured after Jan 2019\P...
Export
An initial (first 1-2 minutes) temperature change of more than 0.1-0.2 C – due to user error - has proven to be the most significant sensitivity of the method.
SAMPLE: SKU2016-CT-1
Measured Data
Particle Distribution
Texture Classes
Soil Triangle
Rarely, but the software may not start logging the time-scale properly. Identify this in the first few minutes, and restart the measurement from the beginning.
SKU2016-CT-1
Pressure
Fit
log10(Time [s])
SAMPLE DATA
Measurement Data
Sample name
SKU2016-CT-1
File name
C:\Users\atne\Dow
Duration
Counter for homogenization
Suspension Data
Volume of suspension
1,000
Particle density
2,65
SIEVE DATA
Particle-size class
Min
Max
Value
Weight
Very Coarse S...
600
2000
2,36
1
Coarse Sand
200
600
2,1
1
Medium Sand
125
200
3,22
1
META DATA
ADVANCED SETTINGS
SOIL CLASSIFICATION
3


METER Pario CONTROL - SKU2016-CT-1
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Settings
Help
PREPARATION 0
IN PROGRESS 0
FINISHED 2
Sample name
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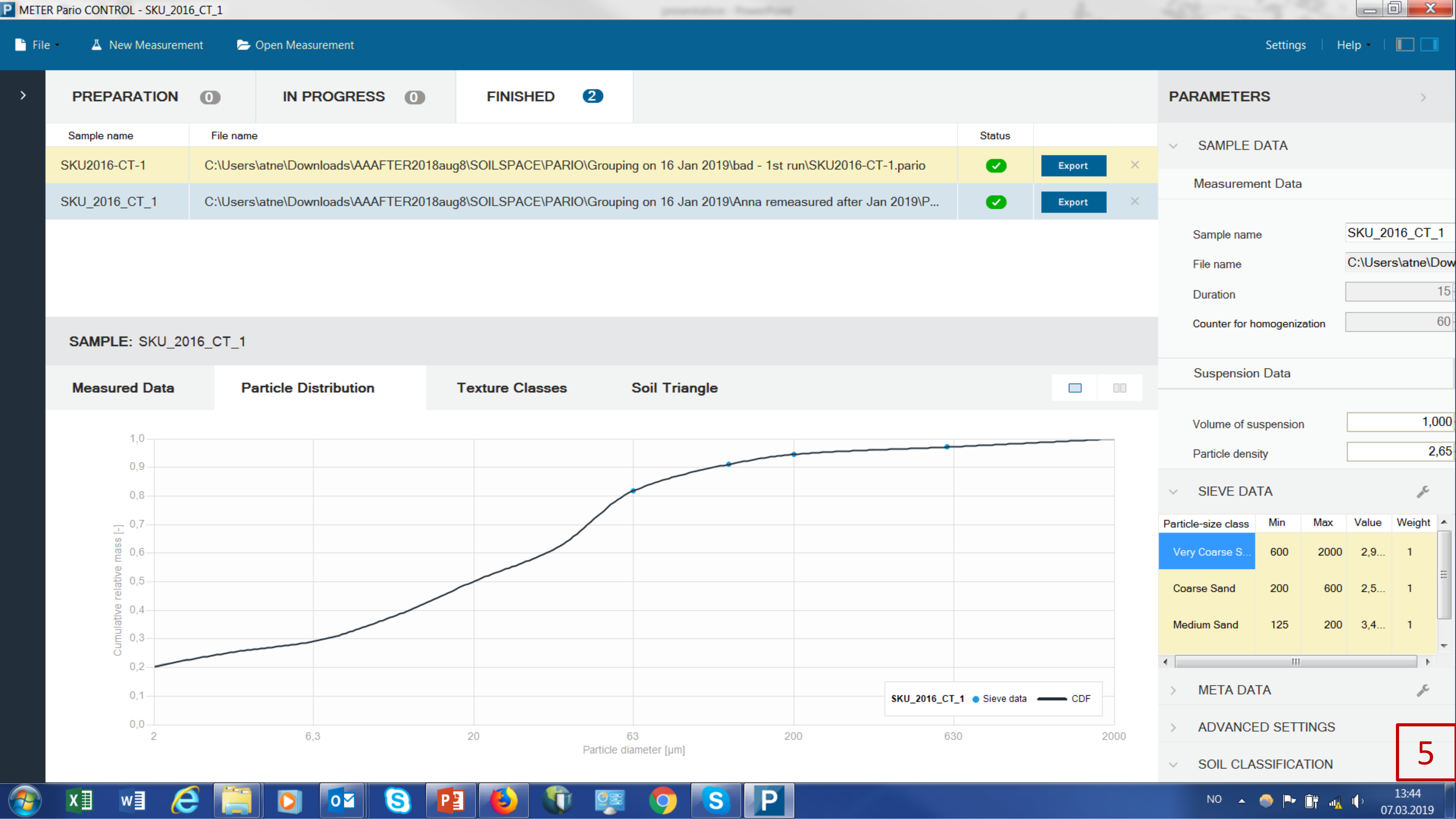
Export
SAMPLE: SKU2016-CT-1
Measured Data
Particle Distribution
Texture Classes
Soil Triangle
Here, there was large uncertainty due to the short run-time. Needs at least 8 hours (vs. recommended 6 hours).
This bump is the consequence of bad early temperature control in the solution. Compare with the next slide that shows the same sample re-measured with good temperature control.
Cumulative relative mass [-]
0,9
0,8
0,7
0,6
0,5
0,4
0,3
0,2
0,1
0,0
2
6,3
20
63
200
630
2000
Particle diameter [µm]
SKU2016-CT-1
Sieve data
CDF
PARAMETERS
SAMPLE DATA
Measurement Data
Sample name
SKU2016-CT-1
File name
C:\Users\atne\Dow
Duration
Counter for homogenization
Suspension Data
Volume of suspension
1,000
Particle density
2,65
SIEVE DATA
Particle-size class
Min
Max
Value
Weight
Very Coarse S...
600
2000
2,36
1
Coarse Sand
200
600
2,1
1
Medium Sand
125
200
3,22
1
META DATA
ADVANCED SETTINGS
SOIL CLASSIFICATION
NO
13:42
07.03.2019


METER Pario CONTROL - SKU_2016_CT_1
File
New Measurement
Open Measurement
Settings
Help
PREPARATION 0
IN PROGRESS 0
FINISHED 2
Sample name
File name
Status
SKU2016-CT-1
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SKU_2016_CT_1
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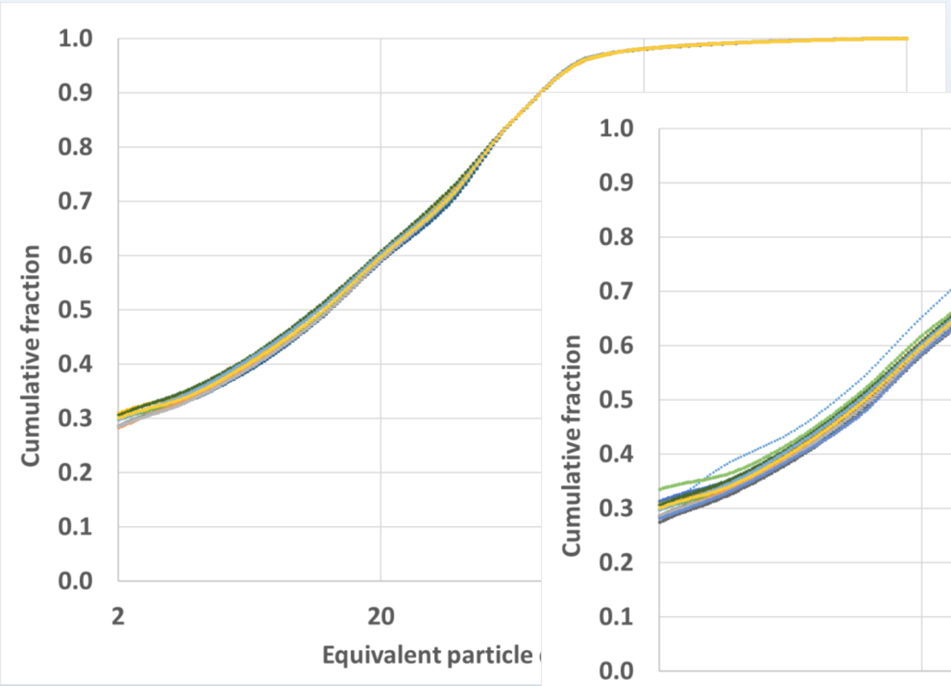
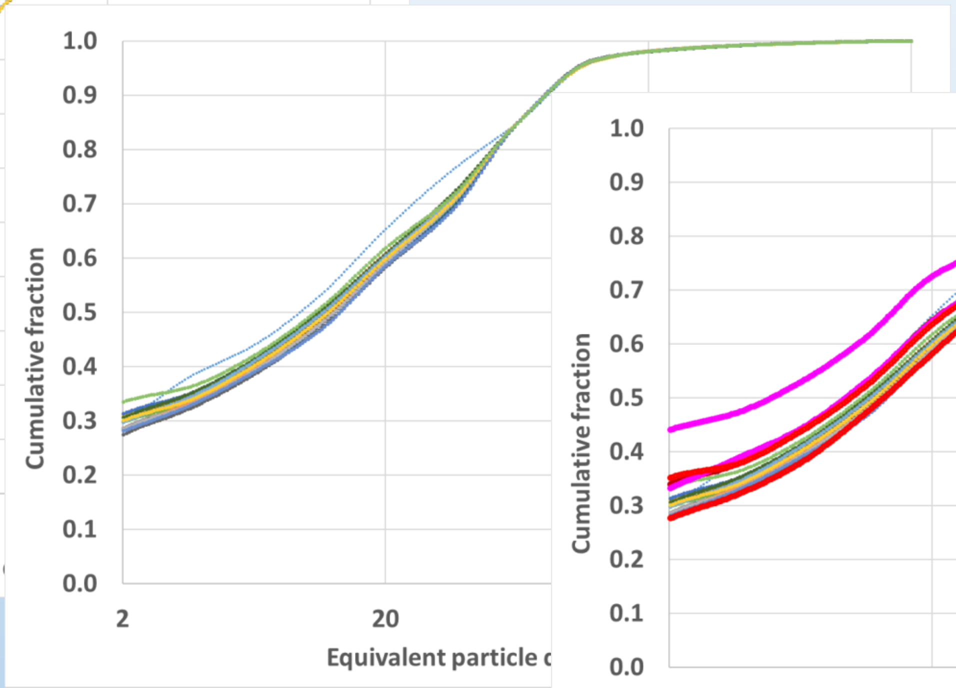
Export
SAMPLE: SKU_2016_CT_1
Measured Data
Particle Distribution
Texture Classes
Soil Triangle
Cumulative relative mass [-]
Particle diameter [µm]
1,0
0,9
0,8
0,7
0,6
0,5
0,4
0,3
0,2
0,1
0,0
2
6,3
20
63
200
630
2000
SKU_2016_CT_1
Sieve data
CDF
PARAMETERS
SAMPLE DATA
Measurement Data
Sample name
SKU_2016_CT_1
File name
C:\Users\atne\Dow
Duration
15
Counter for homogenization
60
Suspension Data
Volume of suspension
1,000
Particle density
2,65
SIEVE DATA
Particle-size class
Min
Max
Value
Weight
Very Coarse S...
600
2000
2,9...
1
Coarse Sand
200
600
2,5...
1
Medium Sand
125
200
3,4...
1
META DATA
ADVANCED SETTINGS
SOIL CLASSIFICATION
NO
13:44
07.03.2019

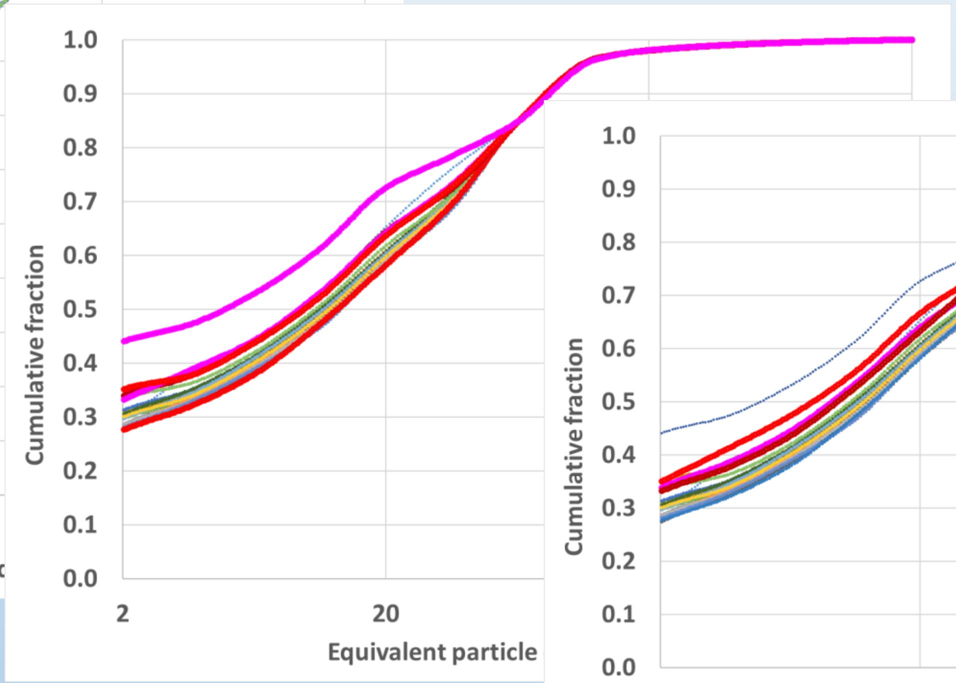
An experiment about repeatability and disturbance by vibration:



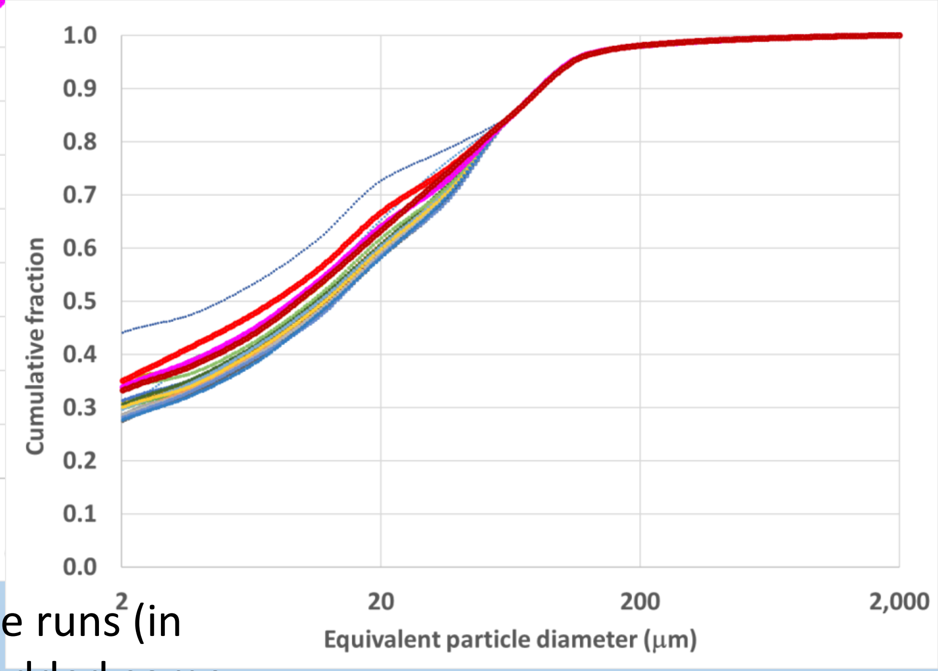
A: May-June 2019:
10 ordinary runs within 18 days using the same solution showed good repeatability



B: May-June 2019:
Including 8 more but *disturbed** runs in the same period showed 'some' extra variability



C: July 2019: adding 5 more runs (in red) on the same column added some extra uncertainty, with one outlying run depicting the magnitude of the effect of the 'initial temperature control problem' introduced earlier.



D: Oct 2019: 3 additional runs after simple re-shaking increased variation just a little (high-lighted in red).

* See next slide on 'Disturbance'

What did ‘*disturbance*’ entail in the above experiment?

- Sample was only physically prepared, sieved, ground in mortar (no chemical pretreatment)
- PARIO rerun 26 times over time in the same re-shaken suspension, each with 12 hours of run-time

- Period 1: May - June 2019:

  10 runs with no intended disturbance

  8 runs with intentional disturbance in the surrounding, as in the adjacent table:

- Period 2: July 2019

  5 runs (no intended disturbance, but one run was affected by a temperature control problem)

- Period 3: Oct 2019

  3 runs (no intended disturbance)

List of possible disturbances - 8 measurements had combinations of these at varied times

Pushed a chair against the table
Recurrently thomped on the table
Banging on the table 1x
Slammed the laboratory door
Bumped the chair against the desk while sitting on it
Moved/slid the measurement cylinder manually
Softly shook the table as when writing on it
Nearby industrial explosion (see footnote)

note: “explosion” refers to industrial rock-fracking by explosives at a construction site some 100m away from the laboratory

Lessons learned along the way:

1. If everything works well, the measurement appears to be well repeatable (both the automated and wet sieving part)

Most significant factors out of those that we tested:

- Vibration: small impact, but it is relative (avoid vibration, but the software bridges small disturbances well)
- **Temperature change over ca. 0.2C in the first 2-3 minutes: large impact (This clearly needs to be avoided! Make sure you temperature equilibrate every hardware and chemical/solution used. Else the temperature effect interferes with the effect of sedimentation on pressure change in the solution.)**
- Sample sitting mixed in for weeks/months: relatively small impact (some flocculation may occur? avoid if you can.)

2. Occasionally the software does not register elapsed time well. This is recognizable in the first minutes, and the measurement can be re-started after re-initializing the sensor in question.

3. Getting your PARIO-based particle-size curve is not effortless. You still do much of the work (sample prep., sieving, data entry) that you do with conventional methods. What you gain is:

- no need for human interaction for 8+ hours
- a continuous PSD curve that can be interpreted in any texture classification systems without interpolation

4. Overall, most of the issues we encountered were user-errors (lack of control), or quickly identifiable and the sample was subject to a quick restart.

What PARIO measurements can solve, and what they cannot:

It helps resolve:

- Compatibility with a large pool of historic data (same theoretical background)
- Classification/Interpolation issues (interpretable according to any 2mm-based systems)
- Provides high-resolution data – necessary for some applications
- Worktime spent with measurement vs. data gained (not too much time saving, but the data gain is large!)

It can/will not resolve:

- Differences rooting from different standards in sample preparation
- Shortening of elapsed measurement time
- Compatibility with laser diffraction data
- It still relies on the basic assumptions that sedimentation techniques depend on (sphericity, etc.)
- The problem that we are measuring >3 orders of magnitude in diameter range with the same one technique

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OK, but how well do PARIO, PIPETTE and LDM measurements actually compare?

(preliminary results)

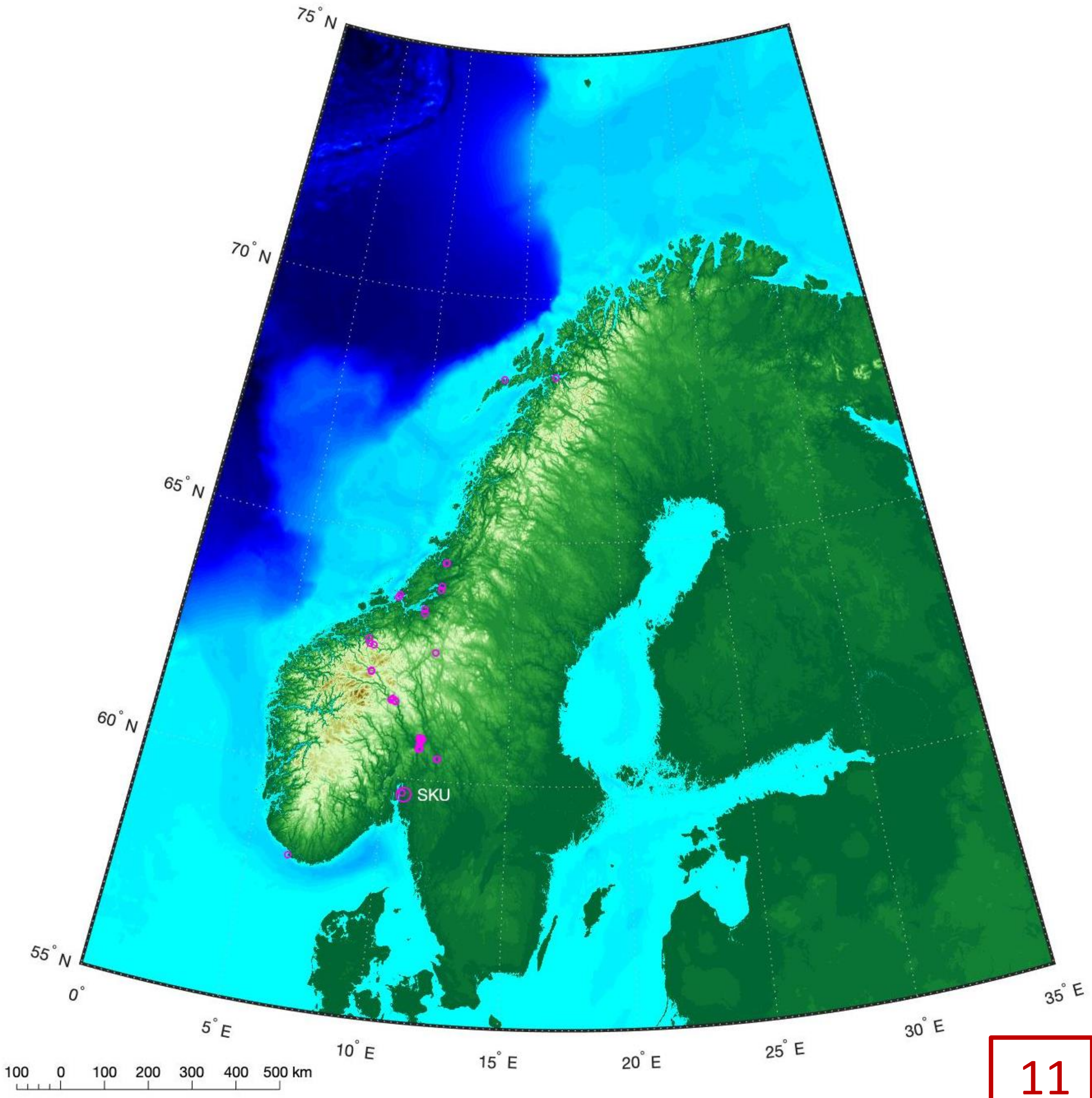
Where did the soils come from?

- 20 samples of varied texture, which is a subset of an N=260 sized new Norwegian data set collected within one project (Soilspace)

The main focus of the SoilSpace project (NFR 2015-2019): *To lay new foundations for the estimation of field-effective soil hydraulic properties using a combination of non-invasive geophysical techniques, X-ray computed tomography, entropy-based mathematical concepts and advanced data mining tools.*

- of mixed parent material (from fine marine sediment to coarse morainic)

- duplicate undisturbed soil cores (200 cm3) were taken for the main project, but also some disturbed sample for the Norwegian Soil Survey program



What samples were used in the measurements?**

- after measuring water retention and hydraulic conductivity on the same samples using a total of 4 methods, the samples were sub-sampled for further measurements (pressure plate, WP4, SOM, PSD).

PARIO: the used soil material originated from 2 separate such cores, and the PSD results were formulated as the average of those two separate measurements

PIPETTE: the used soil originated from the 'disturbed' sample collected in the field adjacent to the 'undisturbed' samples

LASER DIFFRACTION METHOD: the sample was provided from the sub-sample collected from the soil cores (as used for PARIO)

*** the sample collection was not designed for this comparative study, and is acknowledged to potentially introduce some uncontrolled variation*

Sample preparation at a glance:

Summary of the soil samples used:

- 20 samples from Norway
- Subsampling is summarized on slides 11-12
- Sample preparation is described in detail in suppl. material on slides 24-25.

PARIO (by NIBIO's Soil Physics Lab, Norway):

- Sample preparation by the Norwegian standard
- 30g 2mm-sieved soil is used
- H_2O_2 (+ heat) used to burn OC
- HCl is used to dissolve amorphous compounds
- Sodium-pyrophosphate is used as dispersing agent
- Measurement in PARIO's 1L cylinder

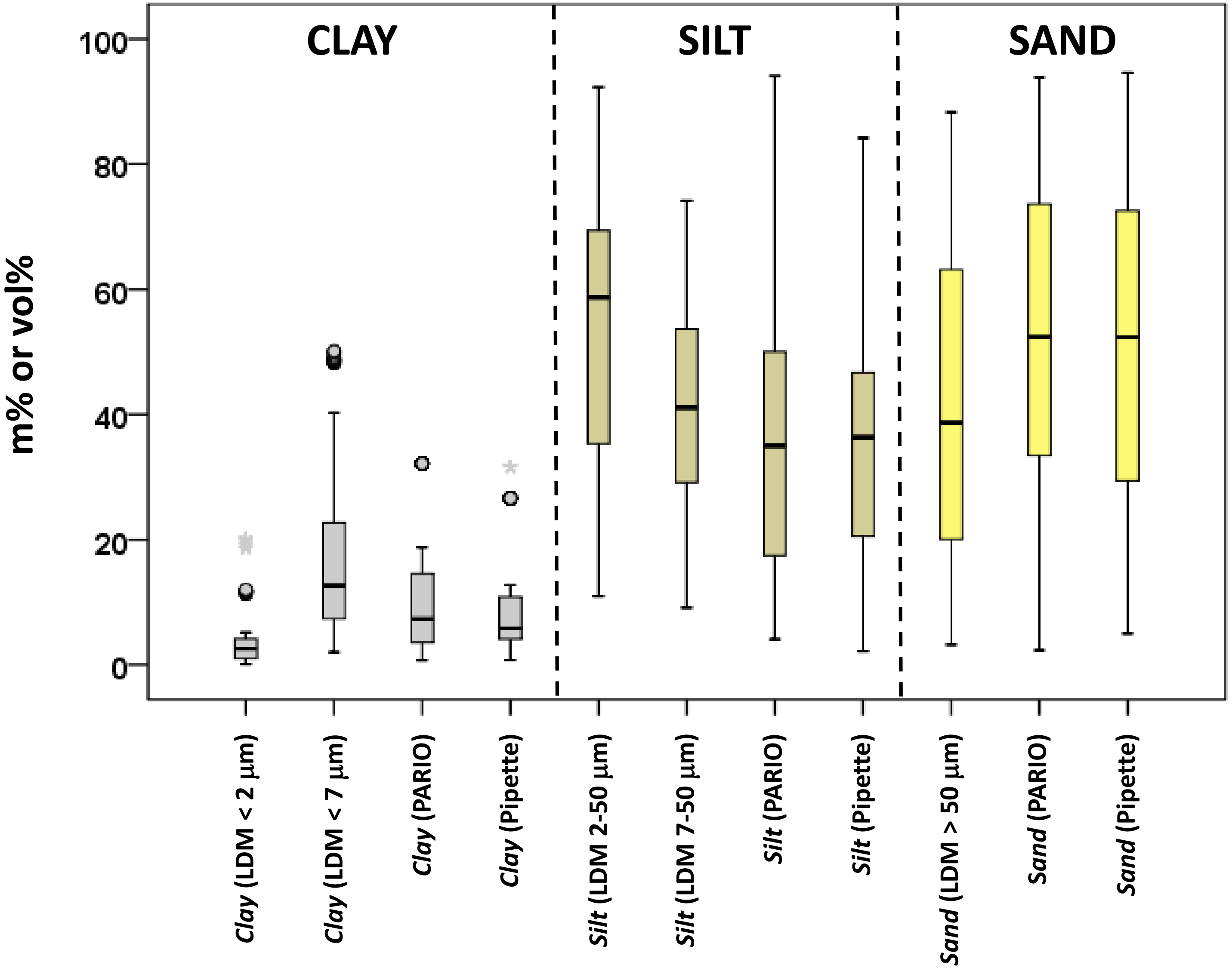
PIPETTE (by NIBIO's Soil Survey Lab, Norway):

- 10g soil was used and 1/3 of each of the chemicals was used in the same process as above with PARIO
- Measurements were done in 400 mL cylinders, altogether 8 fractions were measured.

LASER DIFFRACTION (by the Institute of Soil Science and Agrochemistry, Hungary):

- There was no chemical pre-treatment of samples (no organic matter digestion, lime leaching or iron removal)
- Calgon (a sodium hexametaphosphate and anhydrous sodium carbonate mix) was used as dispersant.

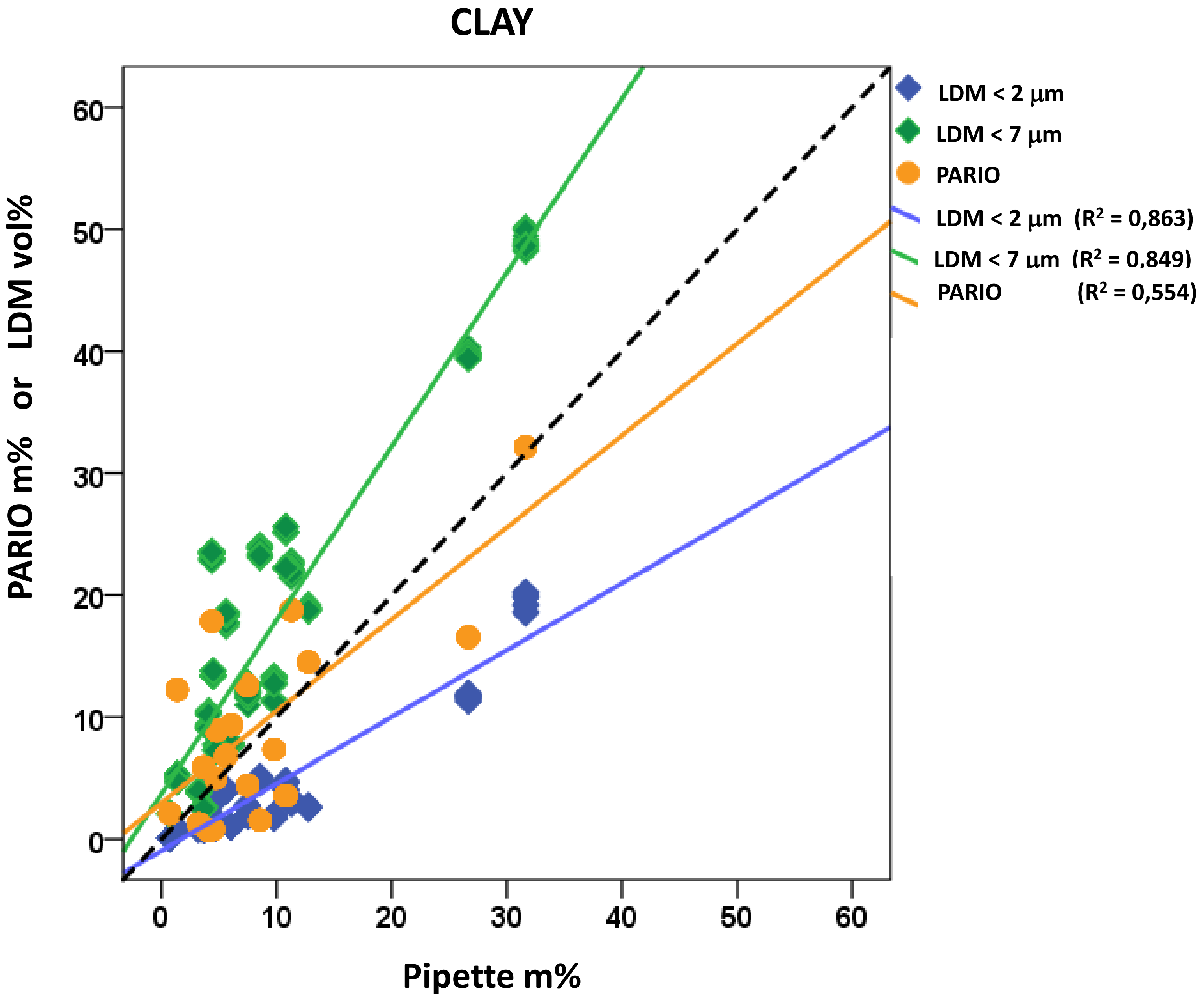
Results (*and discussion on the fly*)



Notes:

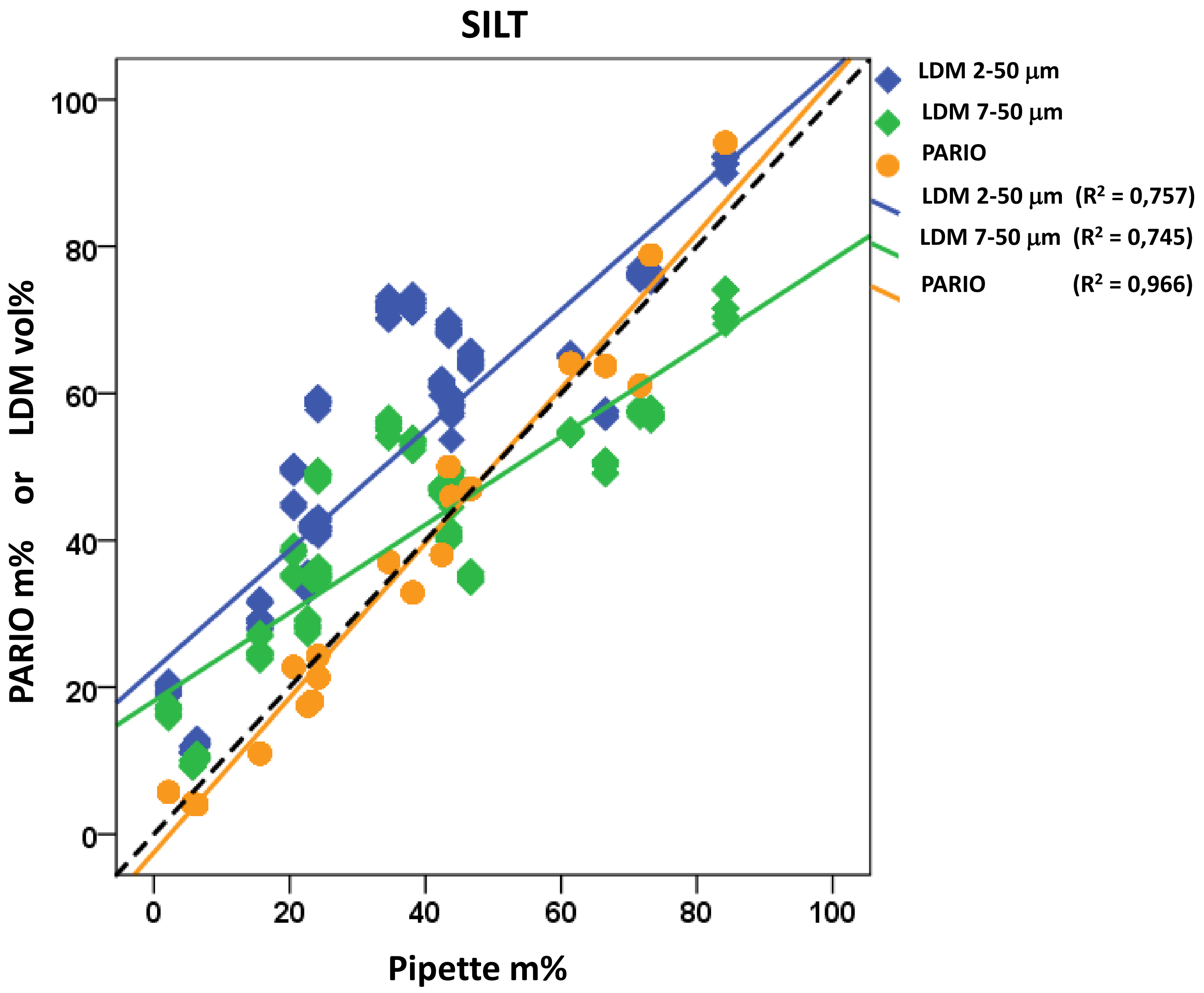
For the laser diffraction (LDM) two alternative 'clay content' equivalents were interpreted as particles either <2 or <7 µm according to *Makó et al., 2019.* (INT. AGROPHYSICS 33: 445–454.)

- For each fraction, the PIPETTE-PARIO populations were most similar to each other.
- For this sample population, compared to the PIPETTE and PARIO methods, clay and silt contents appear to be systematically under or overestimated by the two assumed clay-equivalents respectively by the LDM method.
- The 7 µm LDM clay/silt size boundary seems more appropriate.



Notes:

- The LDM data reflect the previously seen biases in two different directions.
- The PARIO vs. PIPETTE comparison (orange) shows the closest correspondence to the 1:1 line
- There is significant spread in the clay:clay comparisons, reflecting the difficulty in observing the clay (and colloid) size particles.
- The data set is obviously poor in clay-rich samples

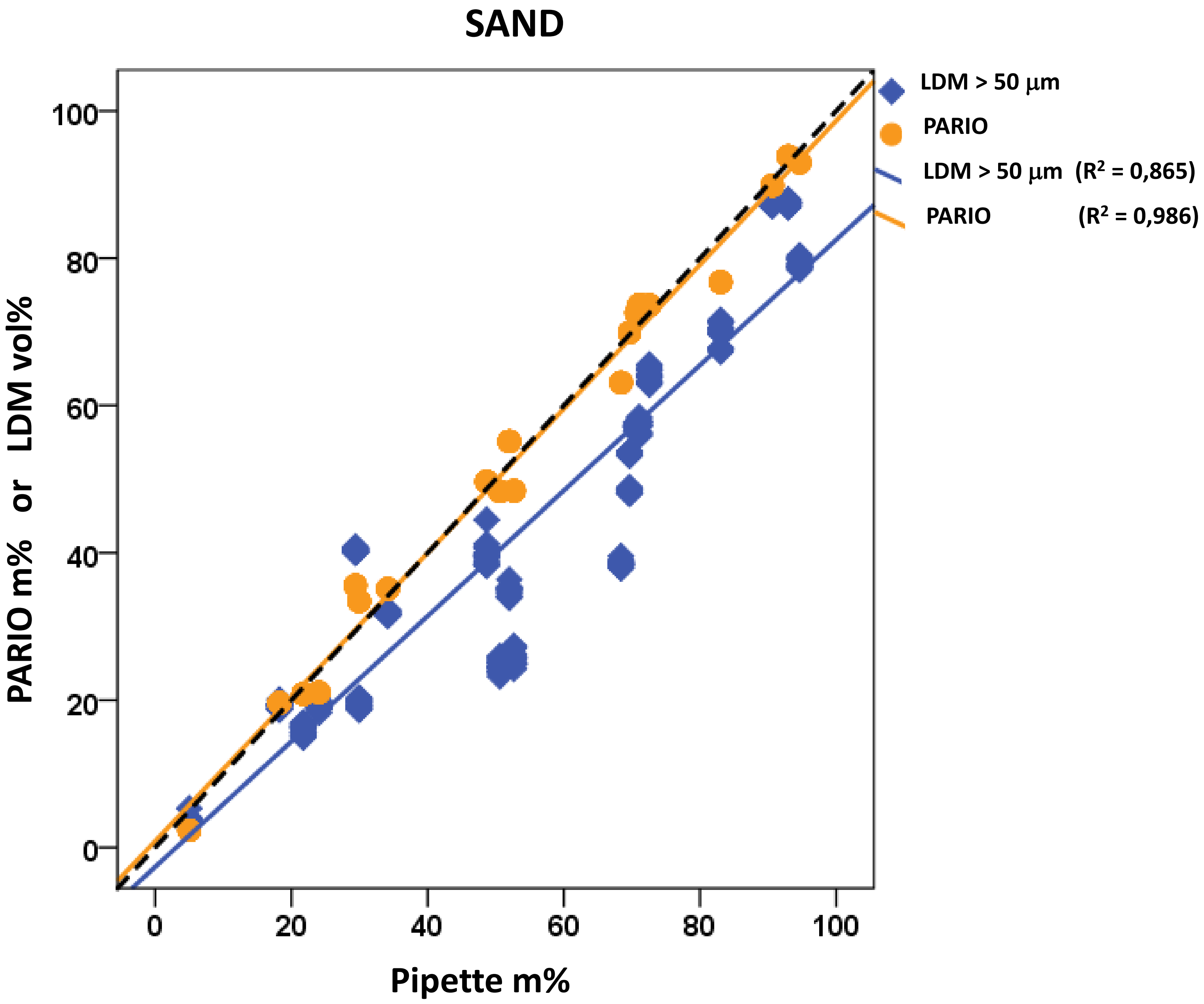


Notes:

- Both LDM derived silt contents show bias relative to the PIPETTE-based silt contents, and this is in relation to the clay content biases, since the two fractions share a size boundary.

- It is likely that for this data set the clay-silt boundary would be ideally established between 2 and 7 microns.

- The PARIO vs. PIPETTE data align very well with the 1:1 line, naturally with some spread in the data.



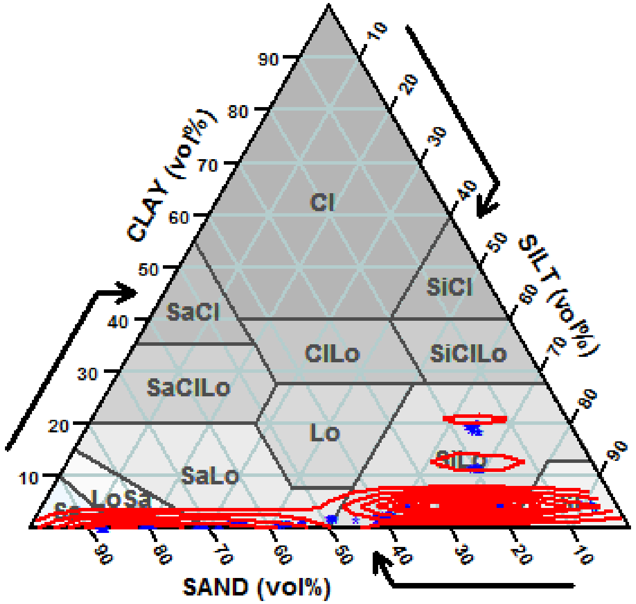
Notes:

- The LDM derived sand contents show some bias relative to the PIPETTE-based sand contents. Their measurement differs a lot, in that the sand content for the PIPETTE and PARIO methods has actually been determined by hand-sieving.

- The PARIO vs. PIPETTE data align very well with the 1:1 line, naturally with some spread in the data. In case of this data set this means that the two different laboratories did a very comparable job in terms of sieving.

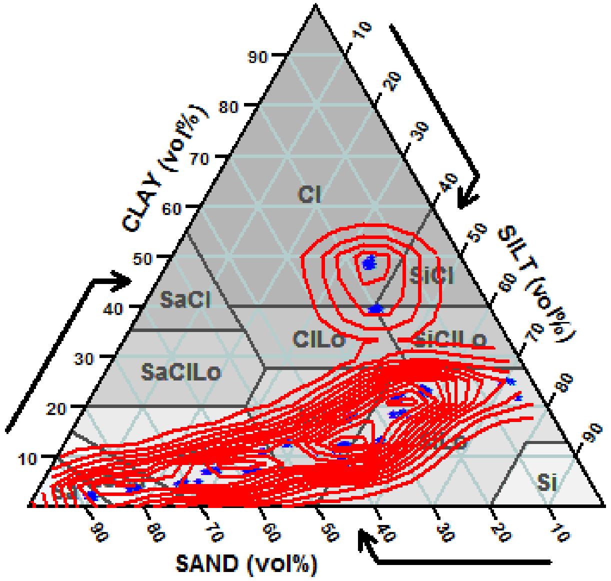
- We remind that the actual samples may have differed a little among the measurements, please see slide 12 for details.

LDM 2.0 micron

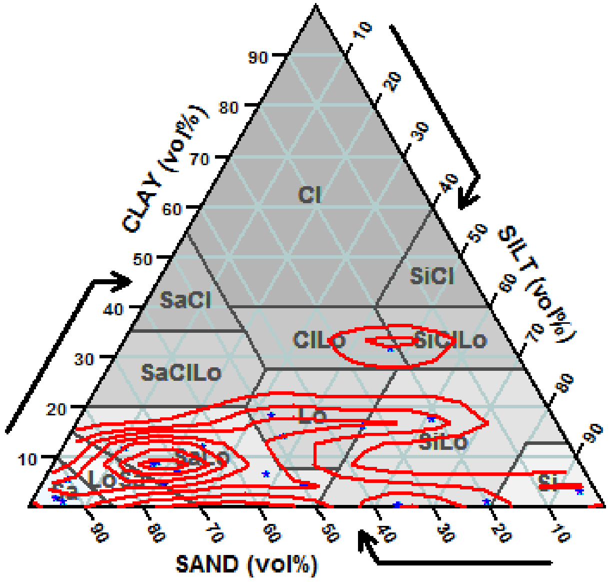


Resulting sample positions in the USDA texture triangle by each of the 4 methods/calculations (with Kernel density map)

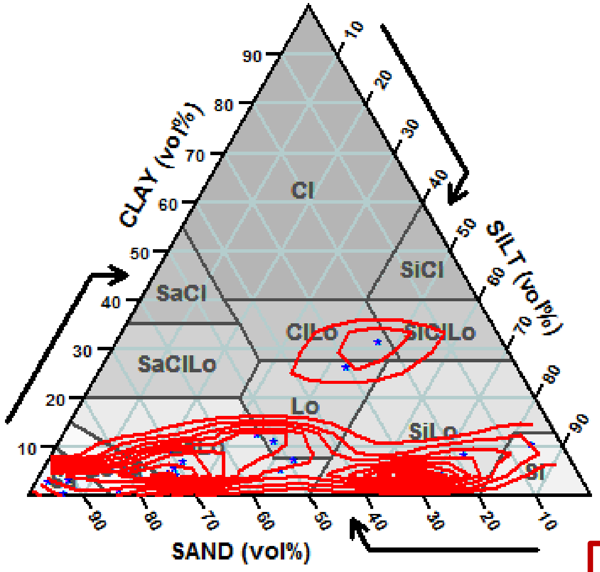
LDM 7.0 micron

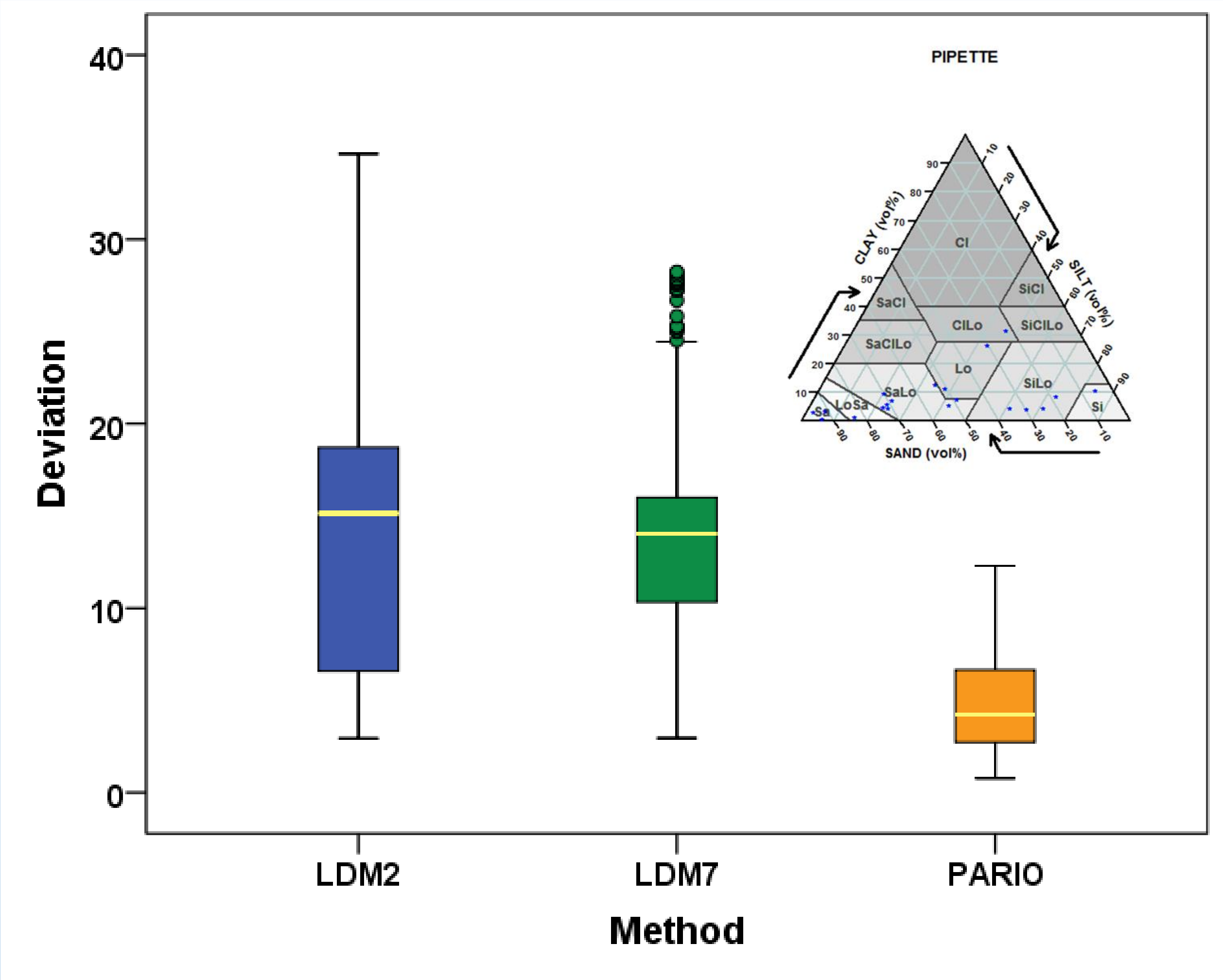


PARIO



PIPETTE



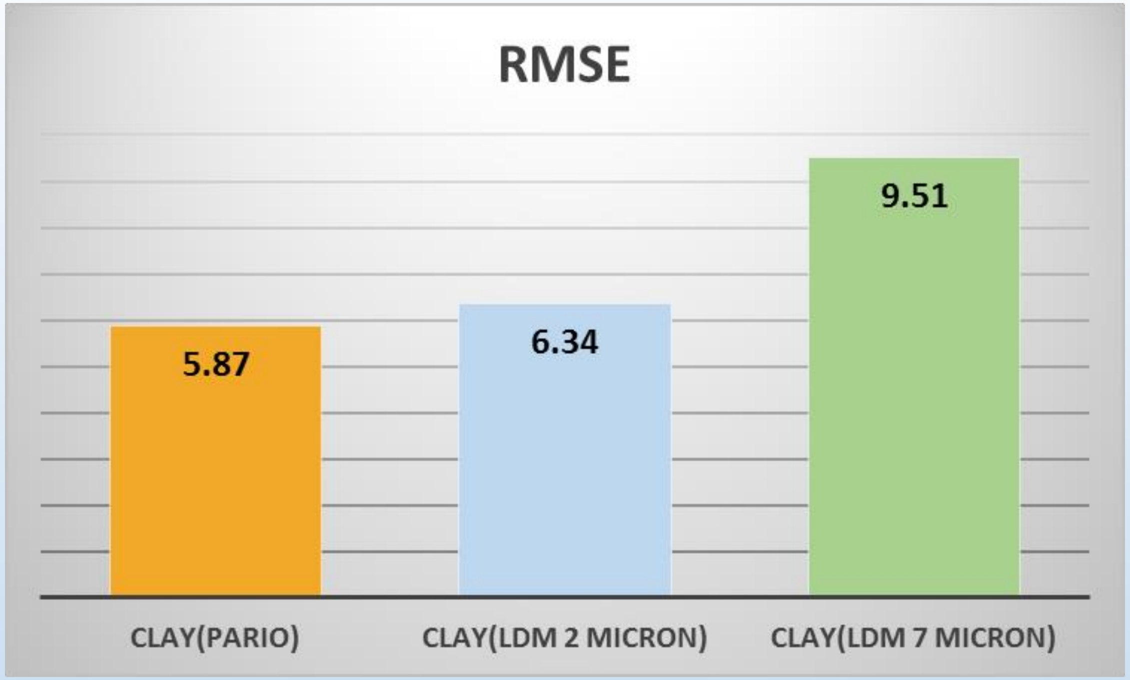


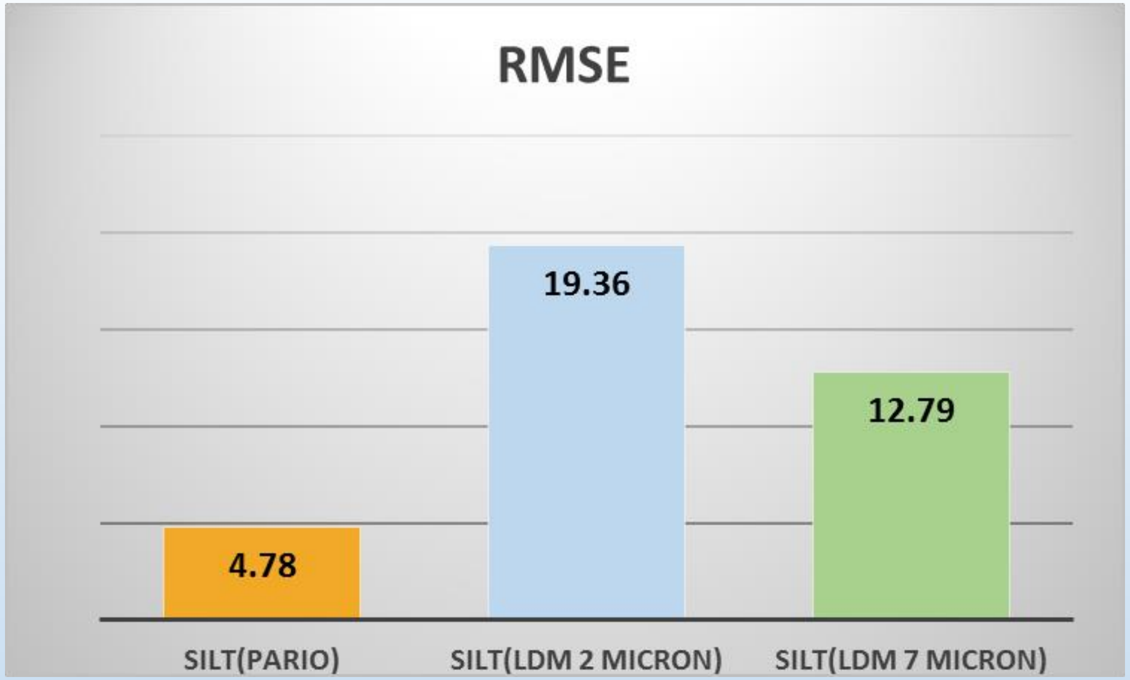
Evaluation of the accuracy of the different measurement methods on the basis of texture triangular diagrams (deviations from the results of the pipette method)

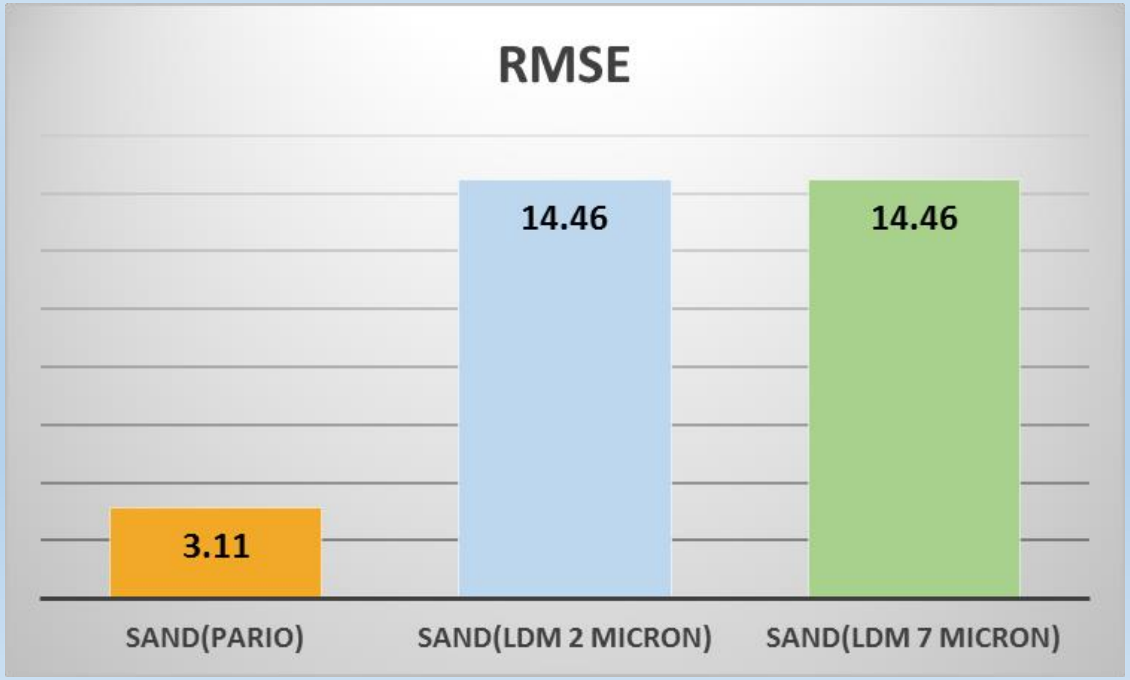
- The particle-size data is compositional (the sum of the particle size fractions is 100%), accordingly an increase in one fraction will certainly lead to a decrease in other fractions.

- Here we converted PSD results into x-y coordinates using the TT.css2xy function of the ’soiltexture’ R package, and calculated the distance between the coordinates obtained for samples using the PIPETTE measurements and the respective other methods.

- Using this dimensionless ‘distance’ variable we evaluated the similarity between the soil textures derived from different measurement methods.





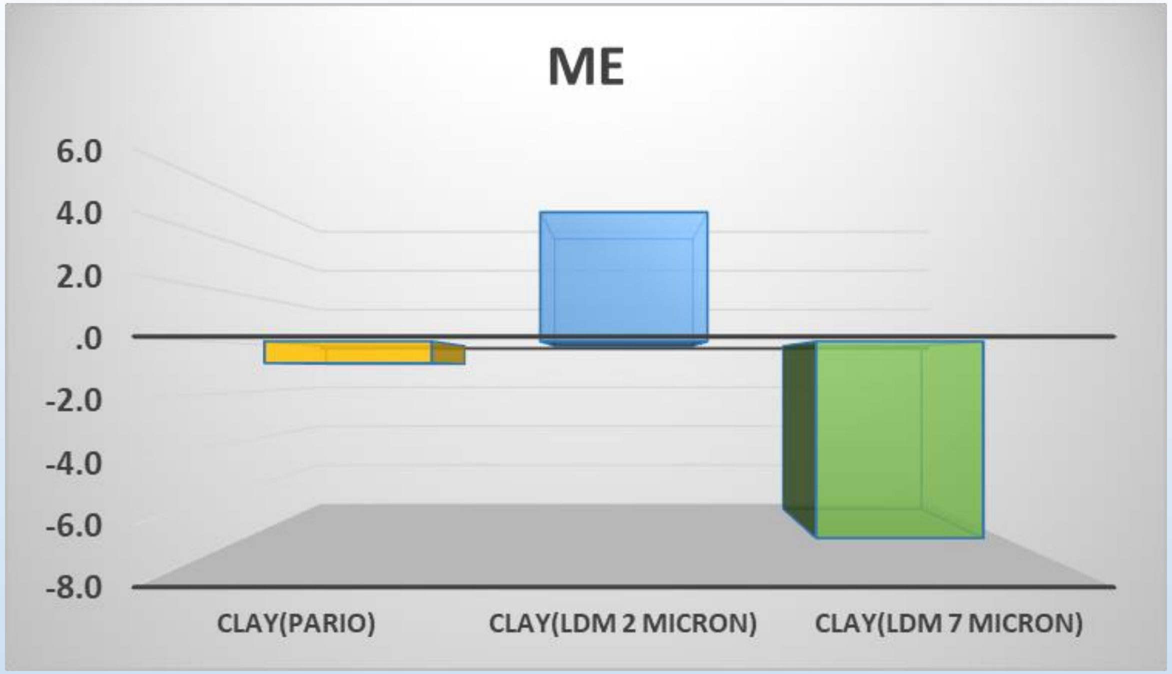


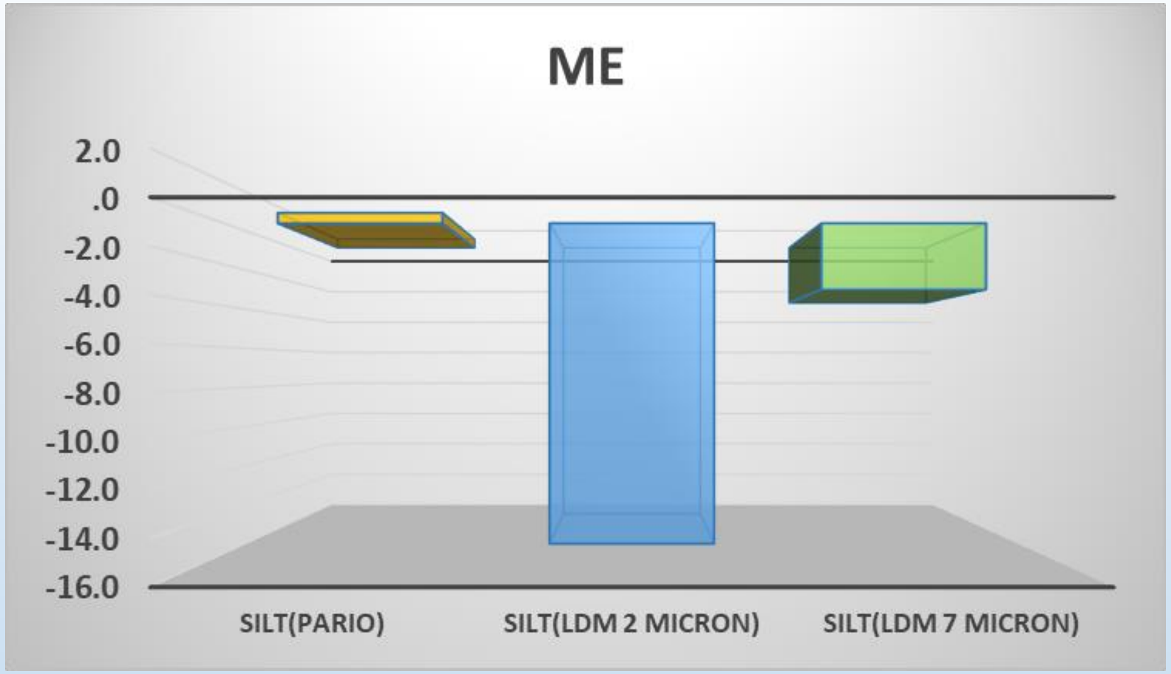
$$RMSE = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(y_i - \hat{y}_i)^2},$$

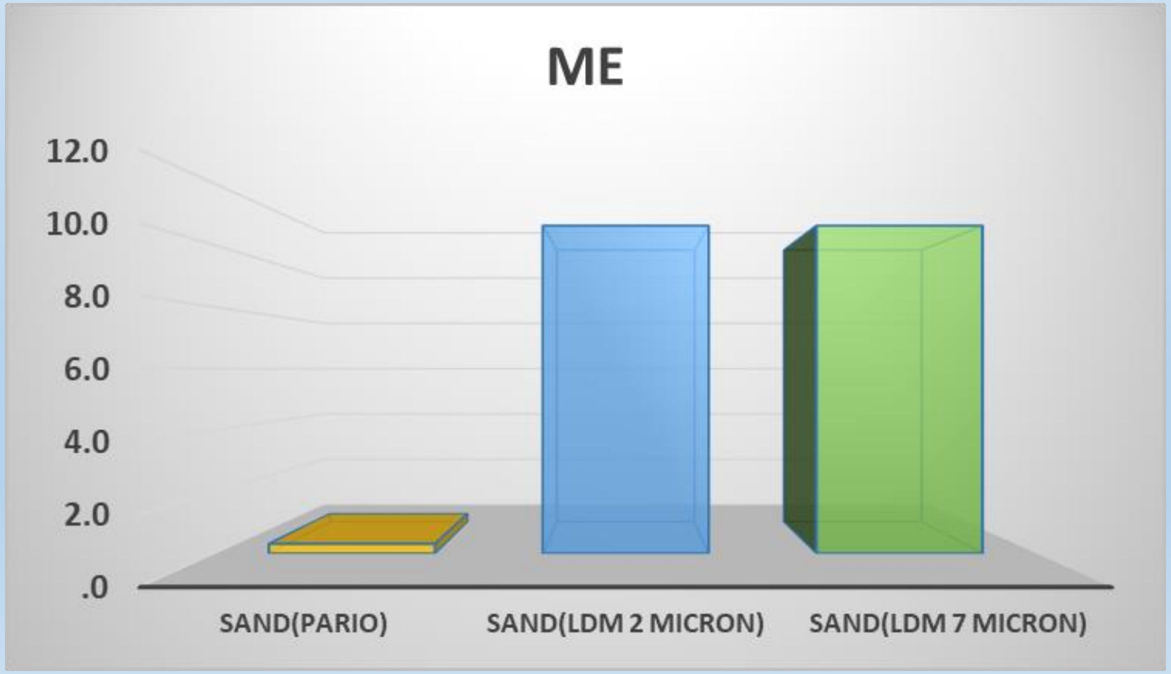
where: y_i stands for the PIPETTE clay, silt or sand values

Notes:

- Texture-wise RMSE-s vary by method and particle group
- PARIO was most uncertain in estimating clay (vs. PIPETTE)
- LDM's performance was dependent on the interpretation used for clay content (2 vs 7 micron limit).







$$ME = \frac{1}{n}\sum_{i=1}^{n}(x_i - y_i).$$

where: y_i stands for the PIPETTE clay, silt or sand values

Notes:

- LDM measurements produced substantial (4-14%) biases per particle group, its direction for clay and silt depended on the applied clay/silt limit (2 vs 7 micron), but it was underestimating sand content (vs. PIPETTE)
- Remarkably, PARIO biases (vs. PIPETTE) remained under 1% for each particle group

Some conclusions, remarks and future tasks

- This was not a planned, targeted comparison! We need to ensure better standardization of the sample among measurements (i.e. homogenized mix to be used). However, we may have emulated a ‘realistic’, practical situation in which sub-samples differ to a certain degree

- Remarkably, while the PARIO vs. PIPETTE measurements show uncertainty – especially in the evaluation of clay content – the biases we found at the sample population level were practically negligible. (*note: METER Group is working on further improving clay estimates*.)

- The laser diffraction method (LDM) returned substantially different data (vs. PIPETTE). Such findings have been reported before. It is noted that LDM does not only differ in its core theory but also in the sample pre-treatment process, as well as the fact that sand content is truly measured together with clay and silt content in LDM (vs. hand sieved separately for PIPETTE and PARIO)

- In terms of PARIO vs. PIPETTE method comparisons, there is a need for additional comparative measurements, especially in soil types and texture ranges that were not represented here. There is a need to fine tune our under-standing of small, but influential differences between the two measurements that may explain part of the uncertainty.

- The optimal conversion of LDM measurements to correlate well with PIPETTE measurements may be data-set or sample preparation dependent, and needs further research.

- It is also a question to what extent the inclusion of sand particles in the sedimentation (and their sieving only afterwards – as in this study) influences the evaluation of clay and silt content

- Let us not forget that all comparisons assume that the PIPETTE method is accurate – which it likely isn’t.

Supplementary material

Sample preparation and notes (PARIO and pipette methods):

PARIO:

- In an 800 mL beaker, 30g 2mm-sieved, air-dried soil is used, 30ml of H_2O_2 is added initially to burn OC, H_2O_2 is topped off as needed (notes: the 30g soil sample includes the sand fraction, which is only sieved after the sedimentation took place. When entering data into the software, the amount of soil was corrected for the independently measured OC content.)
- Ca. 300 mL water was added and heat (ca. 90 °C) applied to burn off any remaining H_2O_2
- 30ml 2M HCl is added to dissolve amorphous compounds, solution is stirred for a minute, then filled to 800 mL and 300 μL of $MgCl_2$ is added to increase sedimentation velocity (*note: Norwegian soils rarely contain any $CaCO_3$*)
- After overnight sedimentation, use a vacuum suction pump to remove the liquid phase as best as possible, then refill to 800 mL and 300 μL of $MgCl_2$ is added again to increase sedimentation velocity. The liquid phase is sucked out again after sedimentation appears complete
- 150ml of 0.05M sodium-pyrophosphate was added and stirred for a minute.
- The sample was transferred to 1L standard glass cylinders and filled up with temperature-equilibrated deionized water.

PIPETTE:

- The amount of soil used was 10g, and 1/3 of each of the chemicals was used in the same process as above.
- Measurements were done in 400 mL cylinders

Sample preparation and notes (Laser Diffractometry):

Malvern Mastersizer 3000, Hydro LV dispersion unit was used. The upper limit of clay fraction was interpreted as either 2 or 7 µm, and the threshold of silt and sand fraction was 50 µm (*Makó et al., 2019*).

Settings: Mie theory, 100% ultrasound treatment for 120 s, 2750 rpm stirring and pump speed, 0.1 adsorption index, 1.52 refractive index of the soil. We used demineralized water during the measurements.

There was no chemical pre-treatment of samples (no organic matter digestion, lime leaching and iron removal). Only dispersion with Calgon was used. The dry soil sample was dampened by a dropwise addition of standard Calgon (33 g of sodium hexametaphosphate and 7 g of anhydrous sodium carbonate in water to make 1 L of solution) dispersant on a watch glass. Thereafter the paste was washed into the tank of the dispersion unit and a further 25 cm^3 Calgon solution was poured on it.

The PSD measurements using LDM were usually repeated nine times: three sample repetitions and three repetitions of measurement per sample.

Malvern Mastersizer 3000

Makó A, Szabó B, Rajkai K, Szabó J, Bakacsi Zs, Labancz V, Hernádi H, Barna Gy: Evaluation of soil texture determination using soil fraction data resulted from laser diffraction method. INT. AGROPHYSICS, 33: 445–454. (2019)