

# Documentation of tests on particle size methodologies for laser diffraction compared to traditional sieving and sedimentation analysis

Department of Geoscience

Charlotte Rasmussen  
Kristian Dalsgaard

Working paper

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CHARLOTTE RASMUSSEN, KRISTIAN DALSGAARD



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Sieving and sedimentation analyses by pipette or hydrometer are historically the traditional methods for determining particle size distributions (PSD). A more informative and faster alternative has for years been laser diffraction (LD). From 2003 to 2013 the authors of this paper have worked intensively with PSD and performed various tests and investigations, using LD, sedimentation (by pipette) and sieving. The aim was to improve and understand the relationship between these various techniques, pre-treatment effects and preferably find a unifying correlation factor.

As a result, method comparisons of LD and sieving/sedimentation are difficult, as LD is a 3D optical volume measurement, sieving is a 2D width measurement, and sedimentation is density dependent. Platy particles like clay are generally measured to be coarser than traditional methods when LD is used. For LD the clay-boundary is found at 6  $\mu\text{m}$ .

For LD, it seems beneficial to split a soil or sediment sample (<2 mm) into 3 fractions at 63  $\mu\text{m}$  and 250  $\mu\text{m}$  respectively. Well sorted samples (like clay, fine silt or windblown sand) can be measured without splitting into fractions, or by splitting at i.e. 63  $\mu\text{m}$ , depending on the clay content and expected PSD.

*Key words:* Soil, sediment, particle size, texture, PSD, sedimentation, clay, sieving, laser diffraction, Fraunhofer, Mie theory

*Authors:* Charlotte Rasmussen ([charlotte.rasmussen@geo.au.dk](mailto:charlotte.rasmussen@geo.au.dk)), Department of Geoscience, Aarhus University, Høegh-Guldbergs Gade 2, DK-8000 Aarhus C, Denmark. Kristian Dalgaard ([geokd@geo.au.dk](mailto:geokd@geo.au.dk)), Department of Geoscience, Aarhus University, Høegh-Guldbergs Gade 2, DK-8000 Aarhus C, Denmark.

# Preface

This publication describes the on-going work of particle size analysis on soil samples carried out by geologist and now lector emeritus Kristian Dalsgaard.

Soils are formed by weathering and erosion of geological parent material. The result is a fragmentation of the solid rock or sediment into smaller particles of amorphous and crystalline (inorganic and organic) material.

Particle size distribution (also known as texture analysis) is widely used in geology and soil science. It is also highly relevant in other industries where it is necessary to know particle sizes and material composition in detail. In earth sciences particle size distribution (or PSD) describes the content of clay (<2  $\mu\text{m}$ ), silt (2-63  $\mu\text{m}$ ), sand (63-2000  $\mu\text{m}$ ) and larger particles such as gravel, pebbles, boulders etc.

Clay plays an important factor in farming and landscape planning. Silt and fine sand influence the soil ability to retain water. Different PSDs within a soil profile could indicate that the different horizons have been formed under various geological events and conditions (Bridges, 1997).

## Nomenclature

PSD: Particle size distribution

RI: Refractive Index

LD: Laser diffraction

# Content

Preface	2
Nomenclature	2
Content	3
Introduction	4
Analytical procedures in the laboratory	6
Laser diffraction (LD)	8
Sedimentation	12
Sieving	14
Removal of organic Carbon	18
Spherical versus non-spherical particles	19
Discussions	25
Conclusions	28
Acknowledgements	30
Appendix 1	31
References	32

# Introduction

Previously, PSD below 2 mm has been determined by combining sieve and sedimentation methods, mainly using the Andreasen pipette or hydrometer. The fraction above 2 mm has not been included because it requires a large amount of sample (Krumbein & Pettijohn, 1938) to provide a statistically valid number of grains. Now the use of other PSD methods and modern instruments raises the need to compare large data sets acquired by old equipment with new data, and if possible, to obtain a way to easily correlate data sets with different origins i.e. by means of a mathematical expression.

The physics of sand transport was developed by Ralph A. Bagnold (Bagnold, 1941). Further investigations have shown that, under appropriate sampling conditions, the distribution of the logarithm of particle size for sands sorted by wind or water can generally be expected to be described by the hyperbolic distribution (Barndorff-Nielsen, et al., 1982). At Aarhus University, Department of Geoscience, the “Water and Soil” laboratory works with PSDs on soil samples and wind- and water transported sediments which are used for descriptions of sedimentary processes and ultimately for soil classification based on the clay<sup>1</sup>, silt and sand content. The basis of the particle size work in the laboratory is a well-defined collection of fourth root scale sieved samples, analyzed on calibrated sieve sets of high quality and described using the log-hyperbolic distribution (Bagnold & Barndorff-Nielsen, 1980) (Christiansen, Blæsild, & Dalsgaard, 1984) (Hartmann & Bowman, 1993). The method of calibrating a sieve set is described later in this paper.

Sieving, sedimentation and hydrometer analyses assume that the particle is spherical. This is not the case with clay particles and minerals like mica which can be elongated or platy (Hayton, Nelson, Ricketts, Cooke, & Wedd, 2001). Clay minerals are hydrous aluminum phyllosilicates with a variable number of cations. Their structure is similar to that of micas, and they form flat, hexagonal

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<sup>1</sup> The term “Clay” covers a particle size range, a group of minerals with negative valence and a soil type. (Guggenheim & Martin, 1995)

sheets. The platy particles play an important role in understanding LD measurements (Pabst, Kune, Havrda, & Gregorová, 2000) .

Clay minerals tend to form complex colloids with humus. Because of these colloids, it is likely that the content of silica, iron and aluminum oxides in soil can form non-soluble aggregates which affect the particle size distribution or cause flocculation (Eshel, Levy, Mingelgrin, & Singer, 2004).

The exact upper limit of the particle size of clay is not universally defined. Most geologists and soil scientists use a particle size less than 2  $\mu\text{m}$ , sedimentologists often use 4  $\mu\text{m}$ , and colloid chemists use 1 micron (Guggenheim & Martin, 1995).

Different soils have been chosen for this study so that they represent a variety of weathering conditions and textures. As described above the content of clay, physical attributes of particles and the humus content are expected to affect the measurements.

## Analytical procedures in the laboratory

All soil samples are initially dried at 110°C for 24 hours, homogenized and gently crushed through a 2 mm sieve. The fraction below 2 mm is used in this study. Organic material should be removed since it results in particles sticking together and, also, organic particles will affect the PSD (like crystalline material, humus has a texture in itself). Organic removal can be performed either by oxidation, using hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), or by performing “loss on ignition” before carrying out a PSD (Carver, 1971).

Peptizers are salts used for dispersion as deflocculating agents, i.e. sodium pyrophosphate (Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>) or sodium hexametaphosphate ((NaPO<sub>3</sub>)<sub>6</sub>). Flocculation is the process where colloids form out of suspension as flocs or flakes that are larger and heavier than the original particles. When using a dispersing agent, the Na<sup>+</sup>-ions are substituted for flocculating ions with high valence, such as Ca<sup>2+</sup> and Al<sup>3+</sup> on the surface of the clay minerals. The result is negatively charged clay particles that will repel each other and split into individual particles (Krumbein & Pettijohn, 1938). The concentration of the peptizer is critical, since a too high concentration will turn it into a coagulant and create the opposite effect (Elonen, 1971).

For sedimentation analysis, the sample is suspended in a solution of demineralized water and a known amount of peptizer. If small particles (and especially clay) stick together, they form agglomerates that react like larger particles in the suspension. During a sedimentation analysis, these agglomerates will not follow Stoke’s law as single particles but react like one or more larger particle – and thus do not settle as expected. The result: The particle size distribution will be interpreted as coarser than it really is. Dispersion itself can be regarded as a fragmentation process (Bittelli, Campbell, & Flury, 1999). The concentration is important: Normally, 20-30 g sample per liter is sufficient, or no more than 15 g clay per solution. Otherwise there is a higher risk of



flocculation (Sørensen & Bülow-Olsen, 1994) . So, it is important to keep a rather low concentration of material and especially clay, keeping in mind you need enough particles for the result to be statistically valid.

Particles finer than 0.1  $\mu\text{m}$  in water remain continuously in motion due to electrostatic charge (often negative) leading to repelling particles. Once their electrostatic charge is neutralized, the finer particles start to collide and agglomerate (or combine) under the influences of Van der Waals forces. Van der Waals forces are defined as the attractive or repulsive force between molecules (or between parts of the same molecule), atoms and surfaces. These forces are relatively weak compared to chemical bonds (Young & Freedman, 1996). Considering the combined effect of Van der Waals forces and electrostatic bonds in a solution of particles in water, the result can be instant flocculation due to the dipolar properties of water molecules (Nielsen, 2000). Clay minerals with shrink-swell capacities like smectite and vermiculite may cause flocculation.

If necessary (and when the sample is dispersed correctly after being shaken well to destroy aggregates), it is easy to split the finest particles from the rest of the sample by wet-sieving at 63  $\mu\text{m}$ . If an additional split is required, the sample is dry-sieved at 250  $\mu\text{m}$  to split the coarse fraction into two fractions (63-250  $\mu\text{m}$  and 250-2000  $\mu\text{m}$ ). No matter how many fractions a sample is split into, the mass of each fraction must always be known for the final data calculations and LD evaluations.

Prior to measurement the wet fractions of the samples (<63  $\mu\text{m}$ ) are dispersed by sonication with an ultrasonic probe.

## Laser diffraction (LD)

LD is described in the international standard ISO 13320 (Jones, 2003). LD is used in material science to describe properties in particle shape, PSD and particle size analysis. LD instruments are fast, easy to operate; they have a high reproducibility and provide detailed information.

Different LD systems use various theories and applications to evaluate the data. Thus, data from different manufacturers may not easily be compared. The diffraction of the laser light results from the interaction of the light with spherical particles and can be described mathematically by Fraunhofer or Mie theory. A Mie evaluation is applicable for pure materials with a known refraction index (RI) and more precise than Fraunhofer on particle sizes up to 10  $\mu\text{m}$  (Mie, 1908). LD systems with different data evaluation software may produce different PSD.

The HELOS LD system (see figure 1) at the Department of Geoscience, Aarhus, is from the German company Sympatec (Rasmussen & Dalsgaard, 2010). The instrument is equipped with 2 main modules: First, a QUIXEL unit for wet samples, consisting of a reservoir with distilled water connected with a quartz flow-cell that allows the laser-beam to penetrate the solution. The flow-cell is available in different sizes. Department of Geoscience has two types: 2 mm and 6 mm. In theory, these two options provide the opportunity to suspend particles up to 6 mm in size (as seen later in this paper this is not recommended). Secondly, a GRADIS (gravitational dispersion) unit for dry samples with a vibrating feeder (VIBRI) attached which allows for particles to fall in a controlled manner through a shaft where the laser-beam passes. Diffraction is measured at the right side of the system by a detector with 31 ring elements.

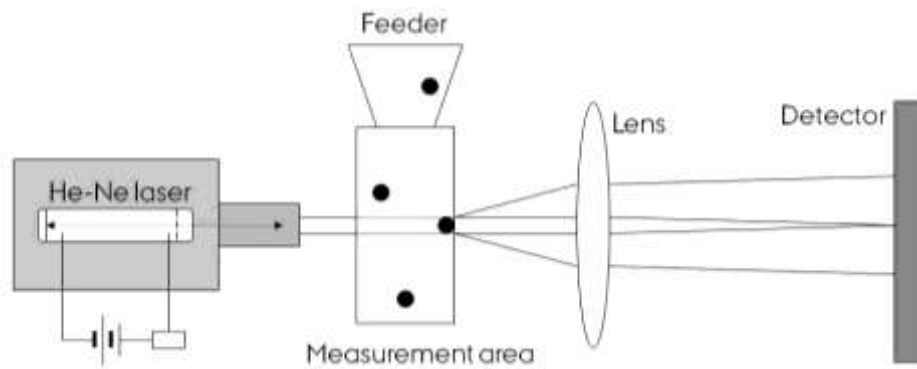


Figure 1: Principle of the Sympatec HELOS LD system. When a narrow beam of monochromatic light is passed through the measuring area containing the sample, the light wave will break when hitting the edge of a particle. The resulting light wave phenomena form the characteristic diffraction pattern. This pattern is focused on a detector, which records the angular distribution of light intensity. **Der blev angivet en ugyldig kilde.** Having measured the light intensity, the size distribution can be found. **Der blev angivet en ugyldig kilde.**

The instrument at Department of Geoscience has three optional lenses. The laboratory has chosen a setup with R1, R4 and R7, respectively where each lens size is a factor 10 larger than the previous. For each analysis, a lens is chosen that covers the expected range of the particle sizes present in the sample. Refer to appendix 1 for the setup of the three lenses.

The accompanying Sympatec software controls all settings during analysis programs, i.e. stirring in the reservoir (a pump ensures random orientation of particles), sonication, duration of analysis and other factors are set up to optimize the measurements to produce high-quality data.

Measurements are normally carried out using the Fraunhofer theory. Evaluating data with the Mie theory is only allowed up to a focal distance of 200 mm (R4 lens) (Sympatec, 2011). Both theories can be used for particle sizes above 10  $\mu\text{m}$ , but the Mie theory gives the most accurate results below 10  $\mu\text{m}$  since the Mie model adds the effect of refraction through the particle and absorption of light. However, certain boundaries and difficulties are to be considered when applying Mie to soil samples. Mie provides good results for fine, highly transmissive, spherical materials and is not recommended for analysis of a mixture of components with different refractive indices or non-

spherical shape. According to Sympatec, the choice of refractive indices can have great influence on the calculated results of the PSD. Further, Sympatec states that the difference between results achieved by the Mie evaluation and results achieved by the Fraunhofer evaluation are almost congruent in the range  $>10 \mu\text{m}$ . For fine, monocrystalline powders Mie theory would be an optimal choice, for instance to be used in the chemical and medical industries.

Sympatec informs that the variance is 1.5 % for coarse particles and 1.0 % for fine particles. The standard deviation is 1 % and the reproducibility "very good" as long as the procedures recommended by Sympatec are followed (Röthele & Puckhaber, 2000). This applies for soil samples split at e.g.  $63 \mu\text{m}$  to avoid interferences from large particles on fine and vice versa. It thus follows that measuring a soil sample on the Sympatec system without splitting ( $0\text{-}2000 \mu\text{m}$ ) the standard deviation will inevitably result in a PSD that shows more coarse particles than is truly present in the sample.

Another benefit with LD: It is (almost) independent of particle density. A small amount of sample is sufficient for a measurement. However, a precondition is that it is representative of the entire sample. Working with very little material may therefore underestimate the coarser part of the sample, since there are very few grains represented during the measurement.

Before each measurement, the signal is re-set by running a reference on the dispersing agent (air or peptizer, depending on the setup using VIBRI/GRADIS or QUIXEL). Measurement is possible with an optical concentration of 5-50 %, preferably 30-40 % according to Sympatec. If the sample concentration is too low, the obscuration and the intensity of the scattered light are low, leading to noisy data. If the sample concentration is too high and the solution too dense, then the light scattered from a particle may be scattered again by a second particle, causing errors in the final PSD

(Bittelli, Campbell, & Flury, 1999). At Department of Geoscience we opt for a concentration of 15-30%.

The applications used ensure that the sample in the QUIXEL unit is mixed while under constant stirring and treated with ultrasonic sound. Ultrasonic sound will degas the solution and prevent clay aggregates to form (Elonen, 1971). The time it takes applying ultrasonic sound should not exceed 60 seconds since some particles with low hardness may break when affected, thus creating a particle size distribution where fine particles are overestimated.

# Sedimentation

Sedimentation occur when particles settle in a suspension. In theory, the coarsest particles (with the largest diameter) will fall faster than the smallest particles. This is described in Stoke's law. The Andreasen sedimentation method makes use of a cylinder and a pipette with a constant volume, where particles with a known density settle in a solution of known volume. The maximum particle size at a certain place in a cylinder can be calculated to a certain point in time (Andreasen, 1939). This method is limited to particle sizes < 50  $\mu\text{m}$  (Krumbein & Pettijohn, 1938) whereas sedimentation by pipette is performed at Department of Geoscience on samples split at 63  $\mu\text{m}$  by wet sieving.

$$v \left[ \frac{h}{t} \right] (^{\circ}\text{C}) = \frac{g2r^2 (\rho_p - \rho_f)}{9\eta}$$

$$t(^{\circ}\text{C}) = \frac{18\eta h}{gd^2 (\rho_p - \rho_f)}$$

v: Velocity of settling particle (cm/s)

$\rho_p$ : Particle density (clay, kaolin and quartz: 2.65 g/cm<sup>3</sup>)

$\rho_f$ : Fluid density (1.00 g/cm<sup>3</sup>)

g: Gravitational force (981.6 cm/s<sup>2</sup>)

$\eta$ : Viscosity at 20°C of fluid (0.01002 poise or g/(cm x s))

h: Height of fall

t: Time in seconds (s)

d: Particle diameter in cm (i.e. 32  $\mu\text{m}$  = 0.0032 cm)

Figure 2: Stoke's law.

Stoke's law (see figure 2) is based on spherical particles and, furthermore, is temperature dependent. Therefore, the analysis is carried out at a constant temperature, in this case 20°C. The cylinders are filled to 20 cm. Assuming that each outtake removes exactly 10 mL (see figure 3), the height of fall h

is 0.4 cm per outtake. To eliminate the assumption of 10 mL, each pipette is calibrated and a correction-factor found. The concentration of the particles has to be held at a reasonable level of 5-10 g per liter (considering the effects described earlier in this paper). Stoke's law assumes that the suspension has a single particle density. Normally the density is set to 2.65 g/cm<sup>3</sup> for soil and sediments originating from parent material like granite and gneiss, yielding particles of mainly quartz and feldspar. However, soil is not heterogeneous and particles not always spherical in the measured range, so this is an estimation (Bah, Kravchuck, & Kirchhof, 2009). Platy particles will not settle like spherical particles: They can cut downwards through the solution in abrupt cycles with pauses very much similar to a leaf falling from a tree.

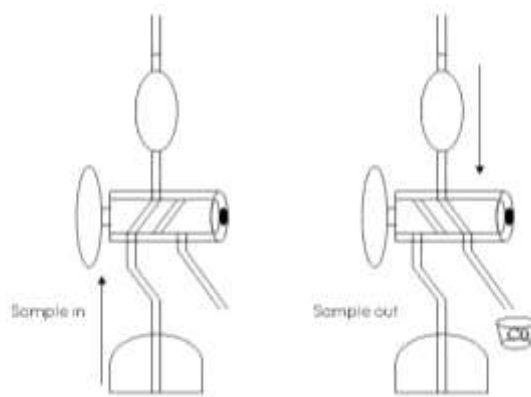


Figure 3: The principle of the Andreasen pipette used for sedimentation analysis.

Turbulence in the suspension must be avoided. Normally turbulence has its origin in temperature fluctuations in the cylinder giving rise to convection currents in the solution. This can be minimized using a temperature-controlled water-bath as described (or vice versa temperature-controlled room). The Andreasen pipette is constructed in a way that allows a small amount of liquid to be retained in the capillary tube which gives rise to a systematic positive error. For some types of PSD, the error is substantial. To avoid this error, the sample residue can be removed before the next sampling. This procedure, however, may lead to another error: Disturbance of the settlement (Allen, 1974), that again affects the sedimentation process and thus the PSD.

# Sieving

Sieving is the process of separating a mixture of particles by size using one or more surfaces provided with apertures of suitable size and shape (Allman & Lawrence, 1972). Sieving (wet or dry) is only possible on rather coarse materials, since the mechanical disintegration is not sufficient to separate very fine particles from each other. A sieving apparatus is relatively inexpensive and the method is highly accurate and precise on spherical materials. When using calibrated sieving equipment, the difference between sieves of various fabricates is eliminated.

Crystalline grains occur in different shapes according to mineral composition, age and transport medium. Two geometrical parameters are used to describe grains: *Roundness* (sharpness, regardless of shape) and *sphericity* (the degree to which the shape approaches that of a sphere). The dimensions of any given form can be described by the three axes: Length, width and thickness (or long, intermediate and short), yielding four different classes of forms. The ratio between the shortest and the longest axis on a particle is known as the aspect ratio. When these two axes are equal in length, the aspect ratio is 1, see figure 4. Particles that have high aspect ratios are therefore not spherical (Zingg, 1935).

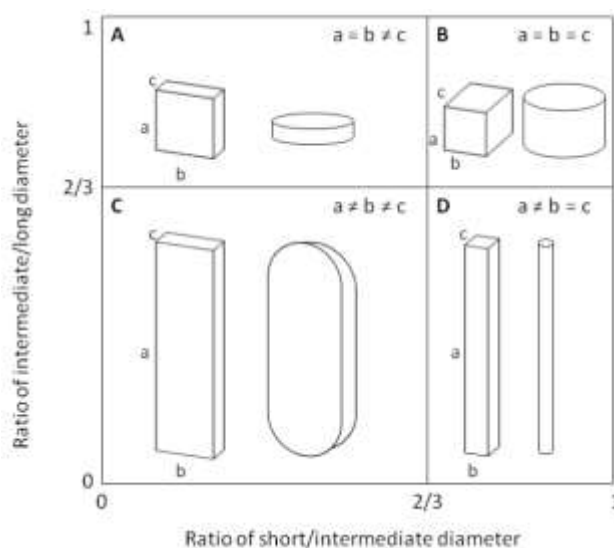


Figure 4: The four classes of grain shape based on ratios of the long (a), intermediate (b) and short (c) diameters. Class A: Oblate (tabular or disc shaped), B: Equant (cubic or spherical), C: Bladed and D: Prolate (rod-shaped). (Zingg, 1935)



Measurements with sieves depend on the sieve meshes yielding the two smallest axes on the particle passing through. Meshes are usually square. A common problem is that non-spherical particles will erect during the sieve procedure, and with their smaller diameter, they will pass through the meshes. Compared with spheres of the same volume, the sieve information overestimates the finer particles. Finer particles usually have to be removed in advance by wet-sieving since fine particles may choke the meshes and prevent other particles from passing. The limiting size dividing fine from coarse is generally between 75 (Allen, 1974) and 105 (Griffiths, 1967)  $\mu\text{m}$ . Normally, 38  $\mu\text{m}$  is used to get close to the range covered by sedimentation analyses, however 63  $\mu\text{m}$  is also quite common.

Aperture sizes were formulated based on a ratio of the square root of 2, meaning the aperture width was doubled at every other sieve in the series. This was later changed to the fourth root of 2, where the aperture width doubles at every fourth sieve instead, see figure 5.

Sieve size: $\sqrt[2]{2}$	Sieve size: $\sqrt[4]{2}$
2.00, 2.83,	2.00, 2.38, 2.82, 3.36,
4.00, 5.66,	4.00, 4.76, 5.66, 6.73,
8.00, 11.31,	8.00, 9.51, 11.31, 13.45,
16.00, 22.63,	16.00, 19.03, 22.63, 26.91,
32.00, 45.25,	32.00, 38.05, 45.25, 53.82,
64.00, 90.51,	64.00, 76.11, 90.51, 107.63,
128.00, 181.02,	128.00, 152.22, 181.02, 215.27,
256.00, 362.04,	256.00, 304.44, 362.04, 430.54,
512.00, 724.08,	512.00, 608.87, 724.08, 861.08,
1024.00, 1448.16,	1024.00, 1217.75, 1448.15, 1722.16,
2048.00, 2896.31,	2048.00, 2435.50, 2896.31, 3444.31,
4096.00	4096.00

$$\sqrt[2]{2} = 1.414214 \text{ and } \sqrt[4]{2} = 1.189207$$

Figure 5: Example of calculating aperture in the range from 2 up to 4096  $\mu\text{m}$ . Note how the value is doubled for every second sieve to the left and every fourth sieve to the right.

Sieving should be performed with a certified set of calibrated sieves and using a standard sieving machine with horizontal rotating and vertical vibration/tapping motion. The sieve set is placed in the machine, and for 20 minutes the grains are jolted at all possible angles, whereby they can align and fall through the meshes randomly. During this procedure, all particles must have contact with the meshes, so no more than 100-150 g should be sieved at a time (Griffiths, 1967). A longer duration may result in coarse particles breaking into smaller particles caused by the mechanical action on the sieve. The sieving loss should be kept below 0.1 % for 30 g samples and below 3 % for the smallest samples of 0.05-0.1 g (Dalsgaard, Jensen, & Sørensen, 1991).

The sieving time depends on a variety of factors, such as the characteristics of the material, sieve size, volume of the charge, relative humidity, and so on. The rejection or passing of particles through the meshes depends on various factors. There is statistical probability that a given particle will present itself at the mesh and fall through. This is determined by the load on the sieve, the particle surface, the dimension and shape of the particles and the method of shaking the sieve. Whether or not the particle will actually pass the mesh depends on the particle dimension and the angle at which it approaches the mesh and tries to pass it (Allen, 1974).

The weaving of sieves cannot be perfect. The apertures are not always square, nor are they of exactly uniform size. As a consequence, PSD determined by sieving often shows a kinky curve when plotted against the nominal sieve apertures. A kink in the curve might arise from a real trait of the particle grading. However, if kinks of the same kind occur at the same size fraction in a series of samples from different localities and with different PSD, it is very likely that the kinks are caused by one or two inaccurate nominal sieve apertures. Therefore, a method of calibrating sieves by means of empirical particle size distributions of hyperbolic shape has been developed. The corrections

found successfully adjust the displacement arising from erroneous aperture up to  $600\ \mu\text{m}$  (Dalsgaard, Jensen, & Sørensen, 1991), see figure 6.

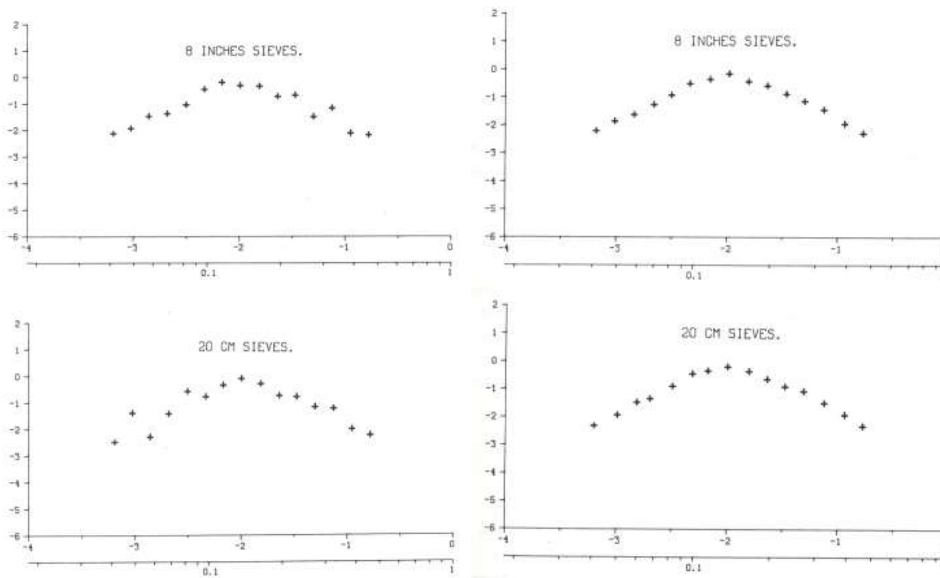


Figure 6: Log-log plots of the size distribution of a marine sand sample (microtidal flat, Mariager Fjord) and the effect of removing the kink on an 8 inch and 20 cm sieve set: Left: Before calibration. Right: After calibration.

# Removal of organic Carbon

The following examples are from the comparison between traditional methods and LD samples (38-2000  $\mu\text{m}$ ) analyzed with the HELOS system on the QUIXEL unit with the R4 and R7 lenses (using the 6 mm flow-cell), and subsequently merged in a single coordinate (100  $\mu\text{m}$ ). Data are obtained from a collaborating project between Faculty of Agricultural Sciences at science center Foulum (in the following referred to as Foulum) and Department of Geoscience at Aarhus University in 2004 (Tøgersen, 2004). 400 samples are from a national soil profile database (KMS, 2002). Another 57 soil standards are from 19 profiles throughout Denmark (Sundberg, Callesen, Greve, & Raulund-Rasmussen, 1999). The traditional PSD results have been achieved by a combined sedimentation/sieving analysis (denoted "traditional" on the graphs). These samples are also oxidized using hydrogen peroxide. Note: Foulum only oxidizes when the content of organic carbon is above 3 %. Some laboratories oxidize only when humus content is above 5 % (Sørensen & Bülow-Olsen, 1994).

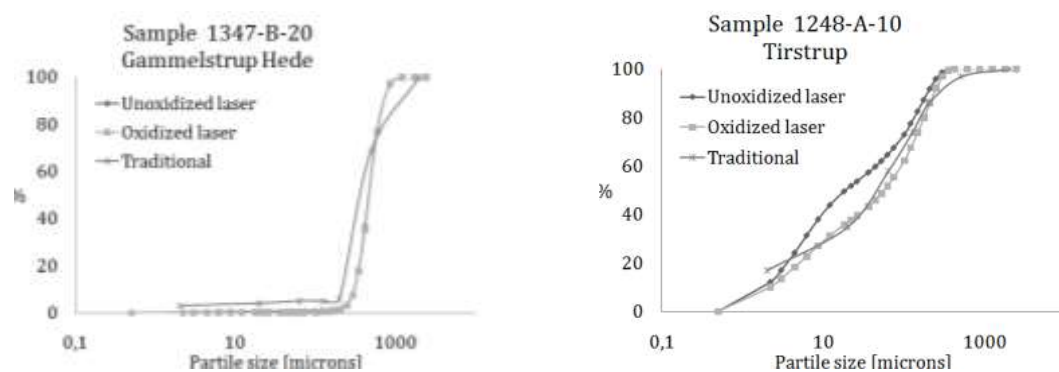


Figure 7: The influence of oxidation on PSD on a B horizon at Gammelstrup Hede (meltwater) with 0.2 % organic carbon and on an A horizon from Tirstrup (till) with 1.9 % organic carbon.

Results in figure 7 show that removing organic carbon from samples that are low in organic material has little or no effect. However, when comparing data containing approximately 2 % organic carbon, it seems clear that removing organic carbon has an effect on the PSD.

## Spherical versus non-spherical particles

Kaolin is used to show how clay particles play a significant role in the understanding of measurements on the LD instrument (Beuselink, Govers, Poesen, Degraer, & Froyen, 1998). The mono-minerals kaolin and quartz have been grinded to ensure that all particles are appropriately separated and within the desired measuring range. Grinding has no effect on the shapes of the particles; it is applied only to reduce the material into its individual components. It has been shown that milled quartz grains are usually angular and somewhat elongated, and occasionally platy. Grinding is no guarantee that 100 % of the grains are non-platy (Beuselink, Govers, Poesen, Degraer, & Froyen, 1998).

The two samples are pre-treated like soil samples. Finally, they are wet sieved at 38  $\mu\text{m}$  and measured on the HELOS system with the QUIXEL unit and R4 (Rasmussen, 2004).

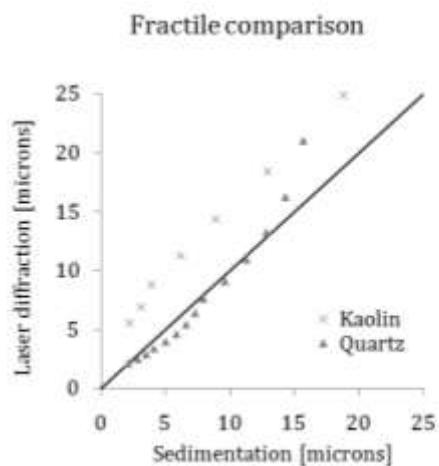


Figure 8: The difference in shape dependence shown on a platy sample (kaolin) and a spherical sample (quartz).

Kaolin shows that 2  $\mu\text{m}$  on the sedimentation analysis corresponds to 5.5  $\mu\text{m}$  on the LD system, see figure 8. Generally, kaolin particles are measured to be coarser with LD than by sedimentation analysis which is very important to note. Quartz shows that the two methods are very similar in the

range up to 13  $\mu\text{m}$ . In theory, the shape dependency should not be visible on quartz measurements, but due to grinding (as described earlier) this is not unexpected.

A clayey till series from Karlslunde has been analyzed. The samples are pre-treated and wet sieved at 38  $\mu\text{m}$ . Additionally, the coarse fraction is split at 212  $\mu\text{m}$  before measurement on the HELOS system with the QUIXEL unit and the R4 and R7 lenses. Sieving has been conducted using the 4th root scale. The sample, originally Karlslunde 710410 (Dalsgaard, 1983), contains visible mica particles.

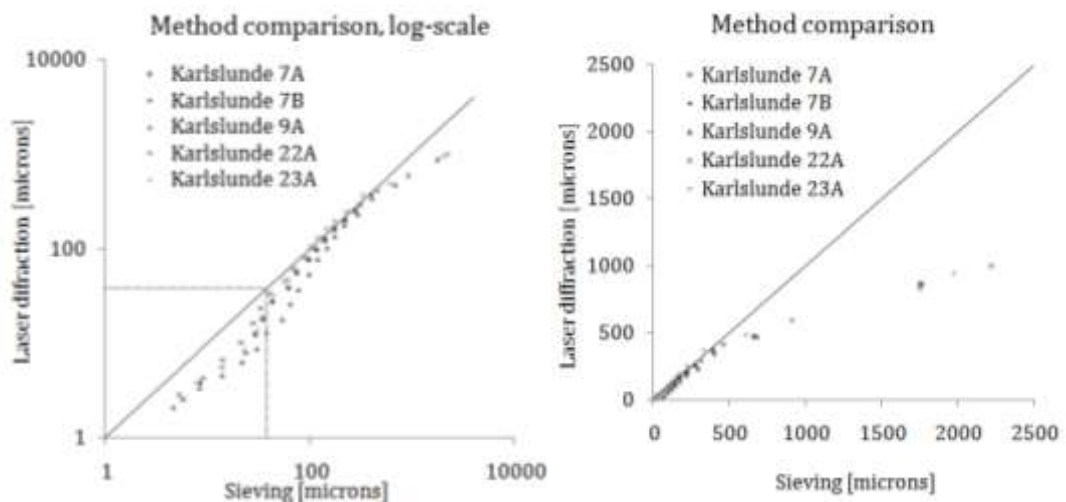


Figure 9: Plots of LD versus sieving. Left is the logarithmic scale, right is a direct plot. The dotted line represents 38  $\mu\text{m}$ .

The data in figure 9 show poor similarity in the coarse fraction above 500  $\mu\text{m}$ . To determine how to measure the coarse fractions (wet or dry), samples from Ribe of coarse windblown sand are selected.

The samples have been sieved using the 4th root scale sieve set.

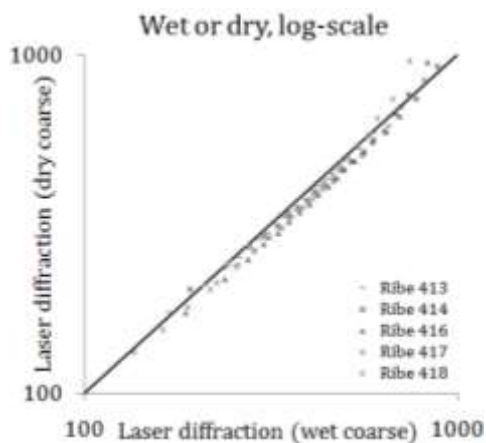


Figure 10: Fractile plot of the five samples from Ribe. The coarse fraction (>212  $\mu\text{m}$ ) with the R7 lens on the QUIXEL unit (using the 6 mm flow-cell), and the coarse fraction (>212  $\mu\text{m}$ ) is run dry on the GRADIS/VIBRI unit with the R7 lens. (Dalsgaard, 2006)

In this case (see figure 10), wet and dry measurements are very much alike. When compared to sieved data, the dry measurements tend to make a better match.

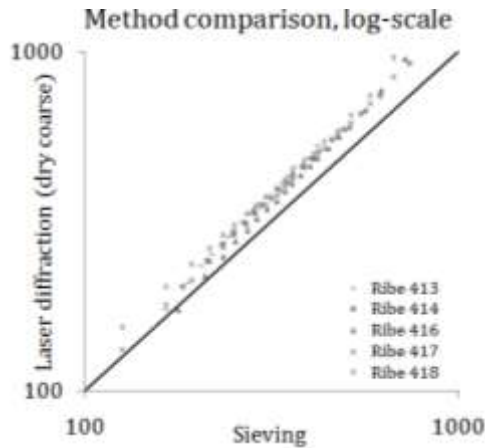


Figure 11: Fractile plot of LD versus sieving of five different samples. The coarse fraction ( $>212 \mu\text{m}$ ) has been run dry using the GRADIS/VIBRI unit with R7. (Dalsgaard, 2006)

Most points in figure 11 are distributed above the line  $x=y$ , meaning that measurements by LD find a coarser PSD than by the sieving method.

The next example (figure 12) is a set of windblown sand samples: Dueodde (Kuhlman, 1957), Kompedal, Ribe 970516-12, Års (Dalsgaard, Greve, & Sørensen, 1991), Stråsø 9 and Stråsø 12 (Dalsgaard and Odgaard 2001) have been analyzed by the sieve-method and with LD. The 20 cm sieve set used has been calibrated, and the aperture has been adjusted accordingly. For LD, the sample was split at  $250 \mu\text{m}$  and the fraction  $>250 \mu\text{m}$  was run with the HELOS system using the GRADIS/VIBRI unit and R7. The fraction  $<250 \mu\text{m}$  was run with the QUIXEL unit using R4 and the 2 mm flow-cell.

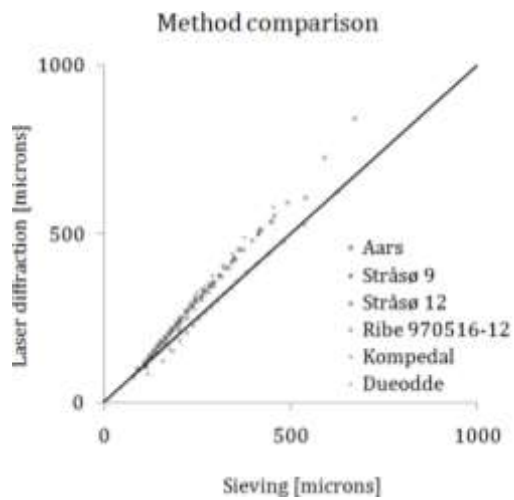


Figure 12: Plots of LD versus sieving on windblown sand samples.

A polynomial calibration between sieving and laser data has been developed by a master thesis student from four of these samples. This equation correlates between data achieved by the two methods, however, limited by the fact that samples must be very similar in mineralogy and texture to the four samples in this test.

$$LD = 2,35S - 87,7 - 0,00221S^2$$

Figure 13: An example of a correlation equation. LD is particle size by laser diffraction ( $\mu\text{m}$ ) and S is particle size by sieving.

The equation is based on the assumption that the relationship can be applied for various other types of samples and that is sadly not the case. Other studies have concluded that fitting performance varies with particle size, not only type and shape of particles (Bah, Kravchuck, & Kirchhof, 2009). So, it seems finding a universal equation is much more complex and dependent on many variable parameters.

Four identical samples from Hestehaven Skov B (clayey till) and six different soil samples from Øster Lem Hede (sandy till) have been measured by the HELOS system with the QUIXEL unit and



R4 on the fraction  $<38 \mu\text{m}$  (using the 2 mm flow-cell) along with sedimentation analysis (Dalsgaard, Baastrup, & Bunting, 1981).

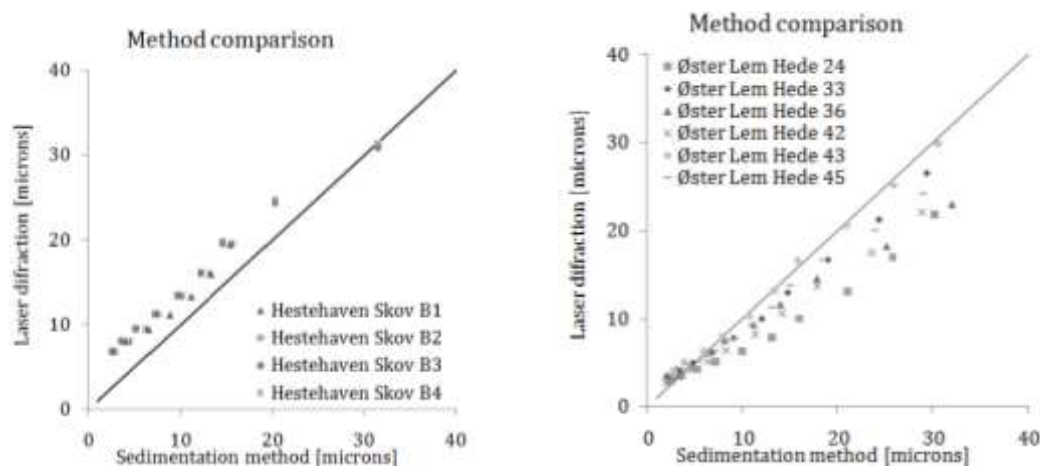


Figure 14: The laboratory standard Hestehaven Skov B, measured 4 times by LD and sedimentation analysis: 1-4 (left). 6 soil samples from Øster Lem Hede (right).

For Hestehaven Skov B, the results of fractile comparison show an almost linear trend  $<20 \mu\text{m}$ , see figure 14. However, LD seems to measure particle size larger than the sedimentation analysis. Especially at the  $2 \mu\text{m}$  limit, the LD measures approximately  $6 \mu\text{m}$ . For Øster Lem Hede, LD seems to measure particle size as being larger than the sedimentation analysis for the very fine material  $<5 \mu\text{m}$ . At the  $2 \mu\text{m}$  limit, the LD measures approximately  $4 \mu\text{m}$ . However, the curve rapidly changes  $>5 \mu\text{m}$ , after which most of the particle sizes measured on the LD instrument are smaller than retrieved by the sedimentation analysis.

To compare different instruments, four particle size analyses were carried out at the University of Copenhagen using a Malvern Mastersizer E series, and at Aarhus University, using the Sympatec HELOS instrument. The Malvern instrument determines PSD between  $0.1$  and  $600 \mu\text{m}$  using Mie theory on small particles and applies Fraunhofer theory for larger particles. Pre-treatment involved dispersion in  $0.002 \text{ M Na}_4\text{P}_2\text{O}_7$  followed by wet sieving at  $500 \mu\text{m}$ . All samples were measured twice

on the instrument (Thomsen, 1999). Subsequently, the four selected samples were measured on the Sympatec HELOS. They were split at 38 and 212  $\mu\text{m}$ , respectively. The fraction 38-212  $\mu\text{m}$  was measured on the QUIXEL module with R4, and the 212-2000 micron fraction was measured on the QUIXEL module with R7 and the 6 mm flow-cell. The fraction  $<38 \mu\text{m}$  was not measured.

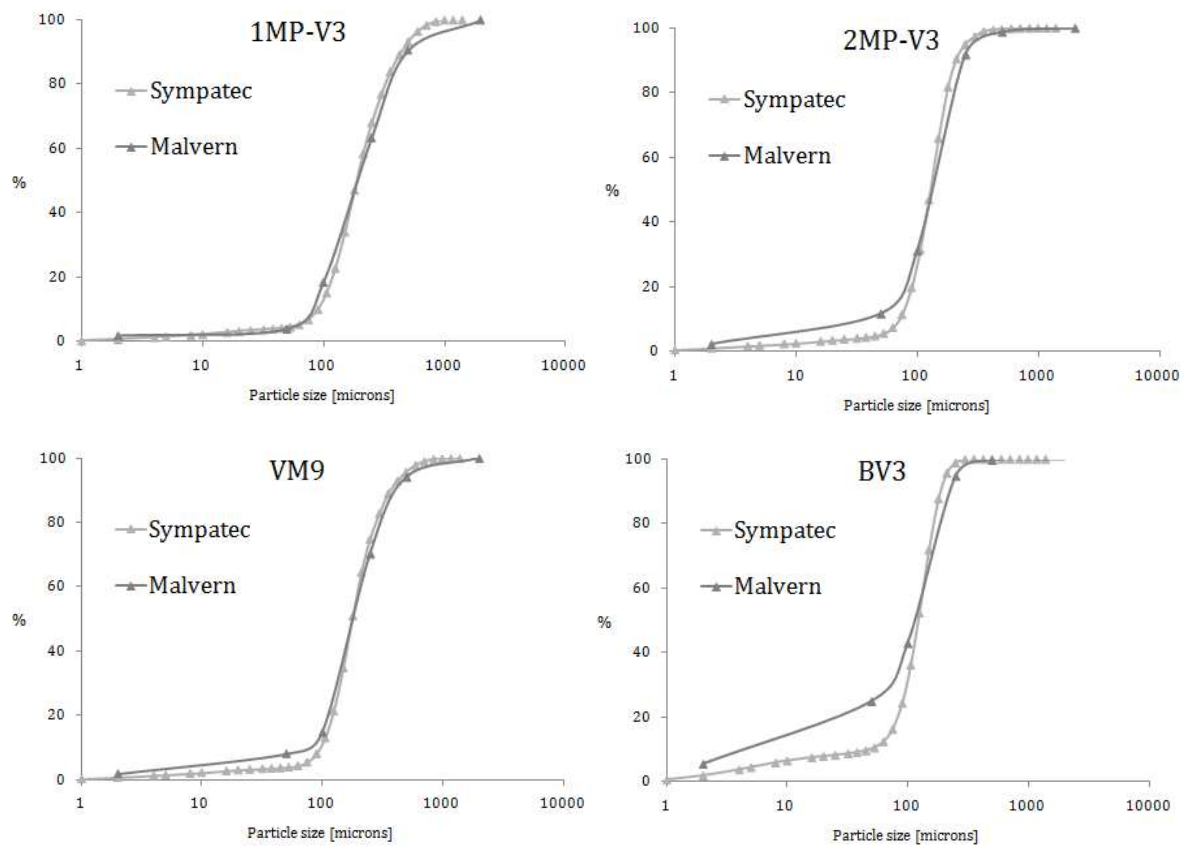


Figure 15: Cumulated values for four samples from Staby (windblown sand) retrieved by two different LD instruments.

Comparing data from the two different LD systems (figure 15), they seem very much alike. The Malvern data sets are achieved without splitting the samples into fractions. This could be a problem when dealing with very fine clay and silt particles that may affect the larger grains when in a suspension. However, given the nature of the samples used here (wind transported sand) this potential error can be disregarded since there is very little material in the smaller particle size range. Any discrepancies in the small particle size range may be related to the different evaluation modes applied by the Malvern and Sympatec instruments.

## Discussions

It has proved to be difficult to derive one equation that describes the relationship between LD results and sieving/sedimentation results. The different methods measure different physical parameters and are therefore not easily compatible, especially when dealing with complex variables like unique shape and mineral composition. Which PSD is the correct one, or is one PSD better than the other? That depends on which attributes the selected method tries to describe. Each PSD may be highly accurate within the fixed limits of the physical properties specified by the method, but “fails” when comparing to other methods.

However, the use of LD technology has many advantages: The method is fast and has a high reproducibility. A wide variety of precise particle sizes can be obtained within few measurements, giving fast and detailed information of the size distribution. The traditional methods like sieving and sedimentation by pipette are easy to learn, but difficult to perform in detail. Results are somewhat subjective to inexperience of the person performing the analysis and the quality of the equipment.

By principle, it is not a good idea to split a sample into fractions using one type of method (i.e. dry sieving), and then run the sample in a different medium (i.e. wet measurements using LD). Variations in the preparatory techniques applied can affect the final PSD of any given sample; therefore, also different laboratory methods will affect data interpretations (Cramp, et al., 1997).

Removal of humus seems to be fundamental in receiving a realistic PSD for samples containing organic carbon. Due to its physical state, organic matter may absorb some light, so the inclusion of organic matter violates one of the basic principles of LD: That all light passed through the

suspension is diffracted (Beuselink, Govers, Poesen, Degraer, & Froyen, 1998). Organic carbon should always be removed, especially if the content is unknown.

Marine samples are not included in this study; however, they will need pretreatment as they may hold a considerable amount of salt that will hinder dispersion and can create flocculation. Samples that are rich in  $\text{CaCO}_3$  should be treated with acid to remove the sticky effect caused by carbonate.

Changing the concentration of peptizer has given no measurable effect. Other studies have found that the PSD depends on the dispersion method, both dispersion agent and the use of shakers (Berthold, Lühmann, Klein, & Nickel, 2000). Changing the amount of sonication prior to measuring has not been tested and should be further investigated in order to make the analysis more efficient. Other studies recommend no more than 2 minutes of sonication. Longer treatment times may cause disintegration of some particles (Blott, Croft, Pye, Saye, & Wilson, 2004) .

The solution provided by Sympatec is to split (especially poorly sorted or bimodal) samples, having three fractions to measure. The Sympatec HELOS setup should be R4 for  $<63 \mu\text{m}$ , R7 for  $63\text{-}250 \mu\text{m}$  (the 2 mm flow-cell in the QUIXEL module for these finest fractions) and R7  $250\text{-}2000 \mu\text{m}$  (VIBRI/GRADIS module). Evaluation afterwards will merge the three fractions considering their individual mass in relation to the total mass of the sample.

The type of merging procedures applied has great influence on the resulting PSD. Especially PSD merged in a single point (as opposed to the Sympatec software's standard "best fit" method) does not take the effect from the fractions into account, which is inconvenient. When using the "best fit" method, each splitting requires the knowledge of the mass of each individual fraction, in order to be able to merge the final PSD.

The content of mica is known to cause interferences during measurement with LD (Hayton, Nelson, Ricketts, Cooke, & Wedd, 2001). When applying sonication and during vigorously stirring in the reservoir, mica may easily break. The mineral also tends to float on the water surface or stick to the sides of the reservoir as well, which was observed several times on the Karlslunde series.

A HELOS QUIXEL setup with the reservoir and the 6 mm flow-cell has issues of keeping coarse particles in suspension. Using the 6 mm flow-cell automatically raises the stirring speed in the reservoir to avoid sedimentation errors. Sorting in the flow-cell may occur anyway since large and heavy particles will plunge towards the bottom. An immediate effect of fast stirring is the formation of unwanted air bubbles the longer a sample is kept in suspension in the reservoir. These bubbles tend to be lead towards the flow-cell and can affect the measurement: The laser light will diffract on the surface of the bubbles and they are measured as particles. At very high speeds, the fluid circulation through the flow-cell can cause particles to orient parallel (rod-shapes particles) or perpendicular (plate-shaped particles) to the direction of flow, making the measurement faulty (Blott, Croft, Pye, Saye, & Wilson, 2004). Therefore, very coarse particles should be run dry on the HELOS system with the VIBRI/GRADIS setup.

## Conclusions

There seems to be no clear or universal relationship between traditional methods and LD. One major reason is the fact that the traditional methods measure mass percentages of particles whereas LD measures volume percentages based on an optical diameter.

Results of LD are affected by variations in morphology and clay content (known as “the shape and size factor”), thus depending on the overall crystalline composition of a given soil sample. The effect is that samples containing clay particles are almost always measured to be coarser by LD than by traditional methods. E.g. a sample determined by the sedimentation method as coarse clay is by LD determined as fine silt. For practical use, LD indicates the limit between clay and silt to be at 6  $\mu\text{m}$  instead of 2  $\mu\text{m}$ . Other studies have concluded the same: That clay particles (below 2  $\mu\text{m}$ ) determined by sedimentation are registered on the laser instrument as being in the size range 5-8  $\mu\text{m}$  (Konert & Vandenberghe, 1997).

For samples like tills, it is necessary to split the sample into several fractions. Other types of well-sorted sedimentary samples (like clay, silt or windblown sand) can be measured without fractionizing. Due to effects while trying to keep large and heavy particles in suspension in the flow-cell, it is generally recommended that coarse particles should be run dry on the HELOS system with the VIBRI/GRADIS setup, and fine fractions can be run wet using the HELOS system with the QUIXEL module and 2 mm flow-cell setup.

Further investigations are needed on the clay-silt boundary and fine silt to clarify the particle shape dependencies, preferably taking particle shape into consideration. This can be performed using scanning electron microscopy and (for coarser particles) another Sympatec product can be useful,

the Qicpic image analyzer (Rasmussen and Mootz 2011). A continuous work is carried out with geologists in Denmark (Callesen et al. 2016).

At the end of the day LD is highly useful to determine particle size, as long as you don't need to compare your data to other methods. However, some preparatory work should still be carried out, like splitting, removal of organic matter etc. to get valuable and reproducible data.

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This working paper is based on the work by several master students and technicians from 1970-2013 at the “Water and soil” laboratory at the Department of Geoscience, Aarhus University. Thanks primarily to the master students Morten Mikkelsen (2000-2004), Katrine Yde Thomsen (1998-1999), Elise Baastrup and Anders Vendelboe (2004-2005), and to the skilled laboratory technicians Bente Rasmussen, Lis Nielsen and Anne Thoisen.

Lone Davidsen and Gry Barfod kindly improved the English.



# Appendix 1

LENS SYSTEM	Measuring size [ $\mu\text{m}$ ] of lens		
	R1	R4	R7
<i>Ring element</i>			
(1)	(0.10)	(0.5)	(0.5)
2	0.18	1.8	18.0
3	0.22	2.2	22.0
4	0.26	2.6	26.0
5	0.30	3.0	30.0
6	0.36	3.6	36.0
7	0.44	4.4	44.0
8	0.52	5.2	52.0
9	0.62	6.2	62.0
10	0.74	7.4	74.0
11	0.86	8.6	86.0
12	1.00	10.0	100.0
13	1.20	12.0	120.0
14	1.50	15.0	150.0
15	1.80	18.0	180.0
16	2.10	21.0	210.0
17	2.50	25.0	250.0
18	3.00	30.0	300.0
19	3.60	36.0	360.0
20	4.10	41.0	410.0
21	5.00	50.0	500.0
22	6.00	60.0	600.0
23	7.20	72.0	720.0
24	8.68	86.0	860.0
25	10.20	102.0	1020.0
26	12.20	122.0	1220.0
27	14.60	146.0	1460.0
28	17.40	174.0	1740.0
29	20.60	206.0	2060.0
30	24.60	246.0	2460.0
31	29.40	294.0	2940.0
32	35.00	350.0	3500.0
<b>Overall range limit</b>	<b>0.18-35.0</b>	<b>1.8-350</b>	<b>18.0-3500</b>

The table shows three measuring lenses available in the HELOS system at Department of Geoscience at Aarhus University: R1, R4 and R7. Sizes denoted (x) are detection limits. (After Sympatec)

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