



PARTICLE SIZING IN GEOSCIENCES

EXPLANATION OF VARIOUS
TECHNIQUES AND PRE-TREATMENTS

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Apart from crystals and broken rocks, not much else in the cosmos naturally comes with sharp angles. While many objects have peculiar shapes, the list of round things is practically endless and ranges from simple soap bubbles to the entire observable universe. Spheres tend to take shape from the action of simple physical laws. So prevalent is this tendency that often we assume something is spherical in a mental experiment just to glean basic insight even when we know that an object is decidedly non-spherical. In short, if you do not understand the spherical case, then you cannot claim to understand the basic physics of the object.

Neil deGrasse Tyson "On Being Round" (Natural History Magazine, March 1997)

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Abstract:

This paper describes different methods for particle characterization, specifically size and shape, from a technical perspective. It emphasizes on analytical procedures in the laboratory, practical challenges and data processing. The content is based on hands-on experience with soil and sediment materials and provides theoretical background information, examples of relevant computations, descriptions of hardware and discussions of using different settings. It is intended to be used as a handbook and may be useful as a guide when designing a local procedure for particle size and shape methods.

To achieve results of the highest quality, attention should be given to the importance of sample handling, relevant pretreatment and dispersion into single, free flowing particles. Equally important is the attention to detail, ranging from proper laboratory working conditions to critical evaluation of data.

One of the main and most important conclusions is the difficulties in comparing data obtained by techniques that measure different properties. This observation does not only apply to particle characterization but is universally applicable.

Keywords:

Soil, sediment, particle size, particle characterization, texture, particle size distribution, ϕ , sedimentation, Stoke's law, flocculation, clay, kaolinite, quartz, sieving, fraction, split, laser diffraction, Fraunhofer approximation, Mie theory, equivalent sphere theory, dynamic image analysis, near infrared spectroscopy, log-hyperbolic distribution, shape, form, geoscience, geology, geotechnical engineering

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The author has extensive experience with analytical methods of soil, water and sediment used for research and teaching purposes within geomorphology, geotechnology and sedimentology. She has specialized in particle size and shape recognition, as well as data processing thereof.

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1 INTRODUCTION



1.1 Background

1.1.1 The use of particle size

In geoscience particle size analysis (PSA) or particle size distribution (PSD) are used as standard procedures for research on soil and sediments within sedimentology, Quaternary geology and geomorphology.

Like most laboratory disciplines, PSA and PSD support the field observations and information already generated and available by other means. Particle size is considered an extremely important clue to the history of geological material: Particle size reveals how the soil or sediment initially was formed and how it has been influenced since formation.

Size distributions contribute to answering questions related to climate, processes and geochemical properties. The following list contains various different areas where PSA or PSD are used:

Agriculture: Silt (and especially fine silt) influences the soils ability to retain water. Thus, particle size information plays a major part in optimizing plant growth, crop health, yield etc.

Construction: Fine and coarse material has been used for thousands of years for building material, ranging from simple shelters to modern-day buildings (clay, bricks, cement). Sand and gravel are used for building processes and landscaping, as well as geotechnical engineering when creating infrastructure such as roads/runways (road base, asphalt), foundations (concrete) etc.

Industrial processes: In industries with milling and grinding, particle size is a critical element in determining efficiency of a process and final quality of a product. Many industrial processes are very much dependent on sand, e.g. manufacturing of glass. Paint is made from powder of a certain texture.

Water: Environmental protection (location of impermeable subsurface clay layers which protects layers below that contain freshwater reservoirs). Hydraulic properties (the soils ability to transport water is dependent on its clay, silt and sand content). Sand is also used in many countries as a filter to purify water.

From this relatively short list it is not questionable that the need for particle size determination has literally been the foundation on which cultures and human civilizations are built. Building material has been used early on to build structures, evidence of this dates back to Mesopotamia (4000 years BC) and the Indus Valley (3300 to 1900 BC). (Nagaraj, Rajesh and Sravan 2016) Sieving is one of the oldest techniques for powder classification: It was used in Ancient Egypt to grade grain during harvesting – and it still is today. (Baert 2019)

So far only few methods have reached and retained a dominant position in industrial application: Sieving, sedimentation and laser diffraction (LD).

1.1.2 The basis of this paper

The author of this paper has worked with research projects and method development, using various techniques for PSA on soil and sediment samples: LD (Sympatec HELOS system), sedimentation (by Andreasen pipette), sieving and image analysis. Most analyses have been carried out at the laboratories at the Department of Geoscience, Aarhus University, in collaboration with scientists, students and visiting researchers.

At the department soil samples are often investigated to describe chemical and biological processes and ultimately for soil classification. Sediment samples are often marine (sea or ocean) or lacustrine (lake) deposits from cores, or till, sand or clay sediments from the Quaternary period. Commonly only material <2 mm is analyzed to determine the amount of clay, silt and sand. These classes represent the most important characteristics and are used to estimate fundamental physical properties.

Sieving and sedimentation analyses by Andreasen pipette have been the traditional methods for PSA of soil and sediments at the department. (Nørnberg and Dalsgaard 2009) A more informative alternative is now LD or image analyses. The choice of technique depends on the PSD and purpose of the analysis.

Note, that there is no "correct" way to represent a size distribution of irregular shaped three-dimensional particles. Each method describes particle size, but it may be by different means. It has

been assumed and accepted that the traditional methods provided the most reliable and “true” particle size information. Maybe a method had its limitations, maybe it over- or underestimated certain size fractions. (Rasmussen and Dalsgaard 2017)

1.1.3 Physical particle properties

In general, comparison between different methods is complex. Especially if methods are based on analyzing different physical properties. Physical particle properties used in PSA are: Geometric properties (linear dimension, surface area/projected area, volume), mass, settling velocity and other types (distortion of electrical, optical or acoustic field). PSA is traditionally known as *texture analysis* when based on mass.

As an example, LD and sieving are difficult to compare: LD is an optical measurement (where the measured area is transformed into volume) and sieving is a width measurement based on mass (figure 1).

Method	Particle property	Quantity
Sieving	Linear dimension	Mass
Sedimentation	Settling velocity	Mass
LD	Distortion of optical field	Area

Figure 1: PSA methods related to physical properties and quantity type.

Unless the material is uniformly shaped, a mono-mineral and exhibits perfect physical behavior (such as fine, windblown quartz sand) it will be impossible to compare measurements directly without creating an advanced model or calibration that suits the exact type of material and specific samples in a batch.

1.1.4 Geology in Denmark

Soils and sediments form 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 (box 1). It is common to include only inorganic crystalline material for PSA.

BOX 1

Rock:

Naturally occurring assemblage or aggregate of mineral grains, crystals or mineral based particles compacted, cemented or otherwise bound together and which cannot be disaggregated by hand in water.

(International Organization for Standardization 2017)

Mineral:

A naturally occurring, solid crystalline substance, generally inorganic, with a specific chemical composition.

(Press and Siever 1998)

Danish soils (figure 2) are well described (Sundberg, et al. 1999) and mostly formed in glacial sediments of Weichselian and Saalian ages. Clayey tills dominate in Eastern Denmark, producing loamy soils (generally 10-25 % clay), while mostly sandy tills, glaciofluvial sediments and windblown sands are found in West-

ern Denmark, resulting in sandy soils (generally <10 % clay). Extensive marine terraces with sandy and silty soils are found in the northern part of the country. (Sørensen and Dalsgaard 2005)

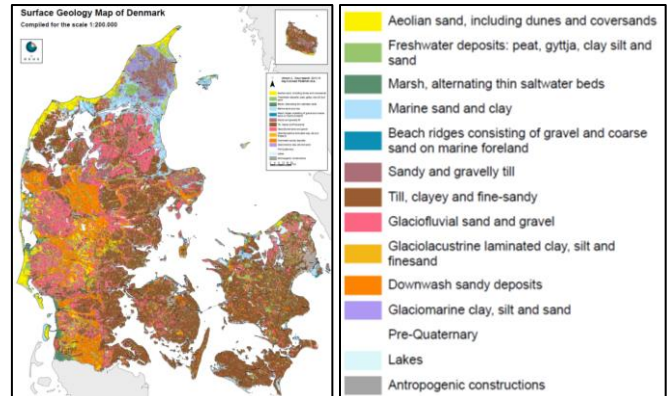


Figure 2: Surface Geology map of Denmark 1:200 000, version 2. (GEUS, 2011/19)

1.1.5 Classification: Triangular plots

Particle size groups (or classes) for soils and sediments range from clay, silt, sand, gravel, pebbles, cobbles up to boulders. Often PSD can be described simply by using the content of clay, silt and sand. For this paper (and unless otherwise noted) the following definitions apply: Clay: <0.002 mm, silt: 0.002-0.063 mm and sand: 0.063-2 mm.

These three classes help determine the soil type and formation. Different distributions within a soil profile could indicate that the different horizons have been formed under various geological events and conditions. Some material may have been transported by river water, some particles are windblown and some sediments deposited by moving glaciers. (Bridges 1997)

When classifying soils the sum of clay, silt and sand is always 100 % and any organic material, carbonates (limestone), gravel etc. is disregarded. Therefore clay, silt and sand can easily be presented on a triangular diagram known as the *textural triangle*, by plotting the percentages of the three fractions (figure 3).

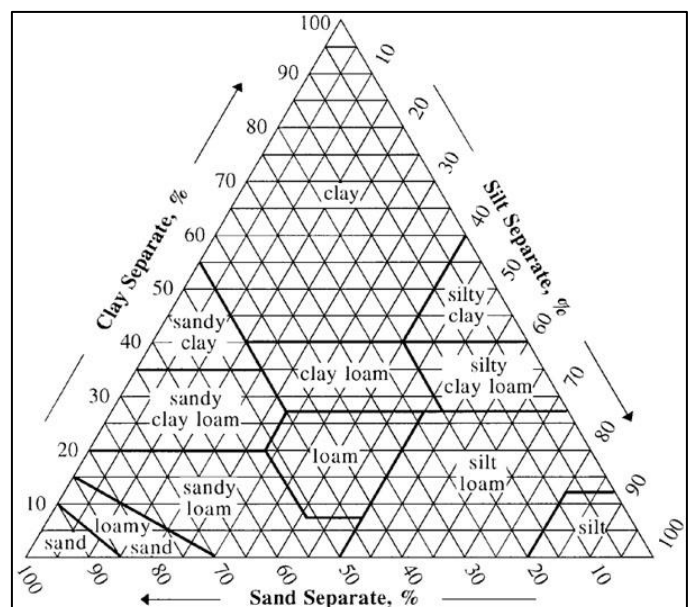


Figure 3: The textural triangle, showing the classification of different soil types. (U.S. Department of Agriculture)

1.1.6 Transportation effects on size

The effects of weathering and transportation largely determines the basic nature of the soil (i.e. the size, shape, composition and distribution of the particles). The environment into which deposition takes place, and subsequent geological events that take place there, largely determine the state of the soil, (i.e. density, moisture content) and the structure (or fabric) of the soil.

Sorting is depending on the density and viscosity of the transport medium. Sorting reflects the transport agent which can be wind, water, ice etc. Usually and over time, particles are reduced in size by weathering and when transported. Meanwhile the sorting will also change: The variation in particle size is reduced. Changes in transport agents and deposition may change the particle size systematically, known as "fining" effects.

Air: Particles are transported by wind through suspension, saltation (skipping or bouncing) and creeping (rolling or sliding) along the ground. Wind will sort particles efficiently because it has low density and viscosity values.

Water: In flowing water, larger particles are deposited as velocity drops, e.g. gravels in river terraces, sands in floodplains and estuaries, silts and clays in lakes and seas. Some currents may have densities close to the particles and a high viscosity. In extreme cases, these currents do not sort the material. In still water horizontal layers of successive sediments are formed, which may change with time (varve), even seasonally or daily.

Ice: Ice has a lower density than water but a higher viscosity. Sorting by ice (figure 4) is ineffective and transport by ice produces particles of all sizes. When grinding and crushing occur, the size distribution becomes wider: Deposits are well-graded, ranging from rock flour to boulders.



Figure 4: Ice is able to carry all sizes of "particles". Here ice has melted and large boulders are left behind. Kangerlussuaq, Greenland.

1.2 Particle size

1.2.1 Units

Particle size is commonly described in the metric system using *mm* (1 mm = 1/1000 m, 1 m is the international unit for length as defined by the International System of Units, SI) or *μm* (1 μm = 1/1000 mm). Refer to the Wentworth grain size chart (appendix A) for different size representations.

1.2.2 φ-notation

The φ (phi) scale avoids names and merely uses numerical numbers to represent size: The φ unit is a logarithmic transformation of size into whole integers (figure 5). This is ideal for statistical

data evaluation. On the φ scale increasing values equals decreasing size. If *d* is a dimensionless particle size, the following relation (Kuhlman 1957) applies:

$$\phi = -\left(\frac{\log(d)}{\log(2)}\right) \text{ or } d = 2^{-\phi}$$

Figure 5: The φ scale. Examples of use in box 2.

BOX 2

Examples of using the φ scale:

φ = 2, then size $d = 2^{-(2)} \times 1 \text{ mm} = 0.250 \text{ mm}$

φ = -2, then size $d = 2^{-(-2)} \times 1 \text{ mm} = 4 \text{ mm}$

1.2.3 Size classes and definitions

Unfortunately, a common problem in science communication is the lack of standardization of some basic definitions, including size classes. Traditionally the clay-silt boundary as well as the silt-sand boundary has not been universally defined. This has been the cause of many errors in academia.

As an example (figure 6), the silt-sand boundary is defined as 0.050 mm by USDA (United States Department of Agriculture) and FAO (Food and Agriculture Organization of the United Nations). However, The Danish Soil Classification is based on the ISSS (International Society of Soil Science or now IUSS, the International Union of Soil Sciences) that defines the silt-sand boundary to be at 0.020 mm. (Breuning-Madsen, Krogh and Kristiansen 2013) Within geology typically 0.063 mm is used as the silt-sand boundary. (International Organization for Standardization 2017)

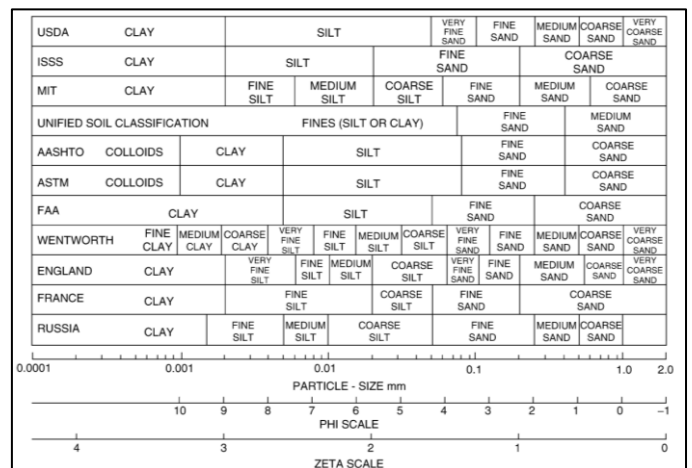


Figure 6: Particle size class limits in several systems. Note that boundaries of clay-silt and silt-sand are often quite different. (Blake and Steinhardt 2008)

Likewise, the exact upper limit of the particle size of clay (box 3) is not universally defined either: Most geologists and soil scientists define clays as having a particle size <0.002 mm. However, sedimentologists often use 0.004 mm and colloid chemists use 0.001 mm. (Guggenheim and Martin 1995)

When discussing clay, silt and sand, it is essential to note what system is used to avoid errors and misunderstandings.

BOX 3*Three definitions of CLAY:*

The term clay covers a **particle size range**, a specific group of **minerals** with negative valence and a **soil type**.

1.2.4 Common parameters

The relationship between different particle sizes and how much (or how often) each particle size occurs is the *frequency* and is described in the PSD: Particle size values are plotted on the abscissa and frequency values on the ordinate as (x, y) coordinates. When evaluating any distribution, graphical representations are interesting to examine. The cumulative curve is often used instead of individual frequency values: On cumulative diagrams all data sets sums to 100 % and the sloping of the curve, peak location etc. enables the interpreter to evaluate relevant parameters of the distribution.

Sorting, mean, median and other common parameters are simple to deduce. Refer to chapter 8 for mathematical equations of the most common statistical parameters as well as more advanced computational options.

1.2.5 Equivalent sphere theory

One basic challenge in PSA is characterizing particle properties using just one number. As described earlier, most particle sizing techniques aim to report PSD using a two-dimensional diagram. However, there is only one shape that can be described by a single unique number, and that is the shape of a sphere. The sphere is the only shape whose properties can be fully defined by a single dimension of length. A sphere of the size "0.1 mm" describes it exactly in three dimensions. If a cube was described as having the size of "0.1 mm", that may describe the length of one edge of the cube, or a diagonal transect of the cube. An irregular (non-spherical) particle can approximately be described as a sphere. Depending on the methods used the resulting size may be very different (figure 7). (Rasmussen and Dalsgaard 2010)

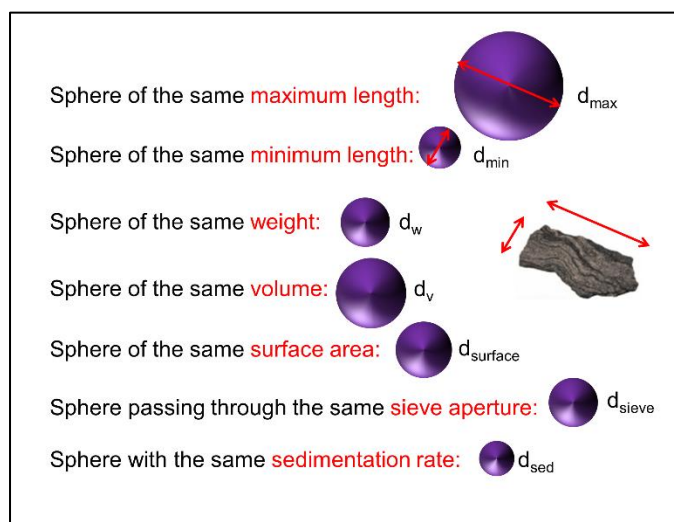


Figure 7: The challenges describing size and equivalents to a sphere.

For this reason, all particle sizing techniques measure the property of a particle and relate this to the size of an "equivalent sphere". One of the most common methods is to measure the

volume of each particle and report the size of a sphere which has the same volume as the particles being measured. This approach applies to LD methods. (ATA scientific instruments 2019)

1.3 Minerals and material

1.3.1 Parent material

Particle size and sorting reflects part of the history of the sediment itself, i.e. the composition of parent material and the weathering conditions (physical, chemical etc.). The degree of weathering is depending on climate and sloping conditions, as well as many other factors related to mineral morphology.

In geology the most abundant and important minerals to know are the rock-forming minerals: Feldspars, quartz, amphiboles, micas, olivine, garnet, calcite and pyroxenes. All of these are influenced differently by weathering and some of them are minerals that are known to influence PSA greatly.

1.3.2 Clay

Many methods for PSD depend on the basic assumption that the particle is spherical. This is not the case with clay particles, nor minerals such as mica: They are all elongated or platy. (Hayton, et al. 2001)

There are four groups of clay: Kaolinite, Smectite (or Montmorillonite), Illite and Chlorite. All four groups of clay consist of silica (SiO_4) and alumina (Al). I.e. kaolinite is built by one layer of silica atoms and one layer of alumina atoms. This structure of one tetrahedral sheet and one octahedral sheet is known as 1:1 clay (figure 8).

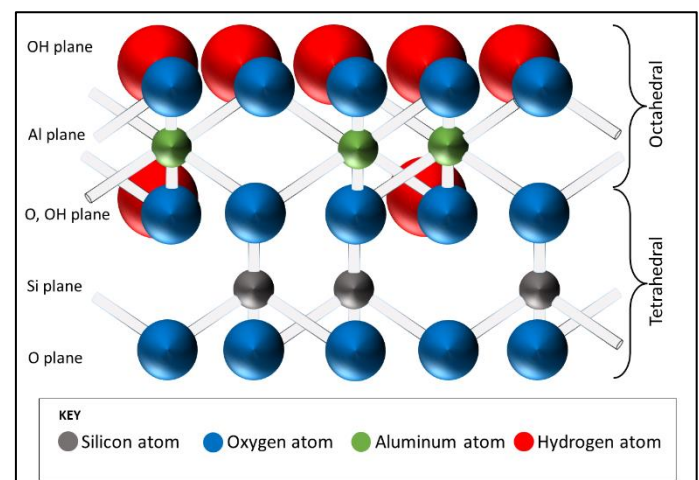


Figure 8: Kaolinite is a typical 1:1 clay mineral. (Bridges 1997)

Clay minerals are hydrous aluminum phyllosilicates with a variable number of cations. Their structure is similar to that of mica: Mica is known to cause interferences during measurement with LD. Mica also has a negative effect on accuracy because of its refractive properties. (Hayton, et al. 2001) Clay minerals form flat, hexagonal sheets. Platy particles play an important role in understanding variations in PSD when comparing between different methods. (Pabst, et al. 2000) Especially kaolinite has often been used to demonstrate how clay particles are measured using LD. (Beuselink, et al. 1998)

Clay minerals usually form by chemical weathering of rocks and soils. Each type of clay has a specific crystal structure. Due to their

tiny size, clay particles often require special analytical techniques such as X-ray diffraction and electron microscopy to analyze their properties in detail (figure 9). Such studies with electron microscope and X-ray diffraction have given clear evidence that many soil clays are made of layered crystals. (Kohnke 1968)

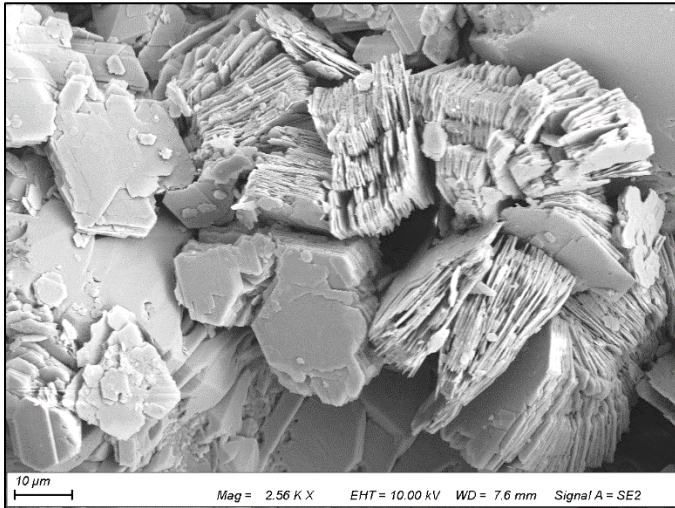


Figure 9: Electron microscopy image showing the platy clusters of kaolinite. Note the size of these clusters are >0.010 mm. (Used by permission from Margrethe Thorup Dalgaard, 2020)

1.3.3 Quartz

Many X-ray diffraction experiments have been performed at the Department of Geoscience on clay as well as the crystal structure of quartz minerals. (S. E. Rasmussen 2009)

Quartz is abundant in soil and sediment samples: Quartz (figure 10) is one of the major rock-forming minerals and on Mohs' scale of hardness, quartz has a value of seven (absolute hardness 100).

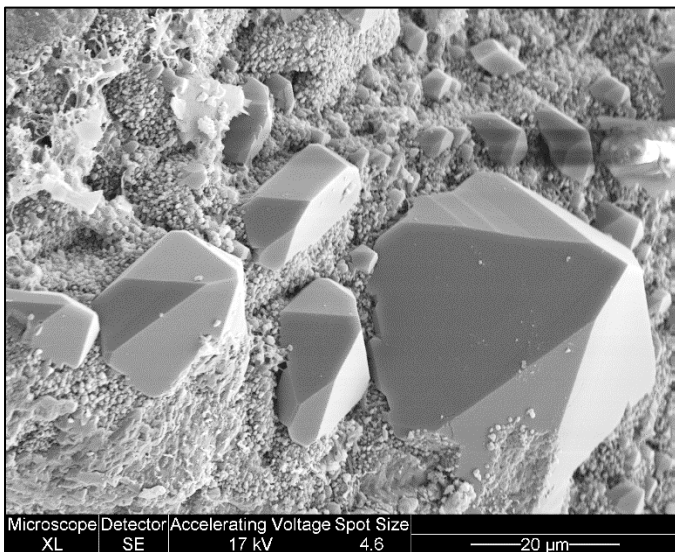


Figure 10: Electron microscopy image showing micro-quartz and larger quartz overgrowths on the surface of a sandstone. (Used by permission from Margrethe Thorup Dalgaard, 2020)

Milled quartz (grinded and carefully sieved) is appropriate to use as a reference or check-material. It is suitable for several PSD techniques including LD. (C. Rasmussen 2004) Quartz has a refraction index (RI) of 1.544-1.553, which is needed for some LD

measurements. Grinding has little effect on the shapes of the particles; grinding is applied to reduce the material into its individual crystal elements. However, it has been shown that milled quartz particles usually are angular and somewhat elongated, occasionally platy too. Grinding is therefore no guarantee that all quartz particles are non-platy. (Beuselink, et al. 1998)

Quartz has the overall formula SiO_2 and is built from a framework of silicon-oxygen tetrahedra (figure 11). As such, quartz particles are often found to be spherical and therefore one of the most perfect structures to carry out PSA on.

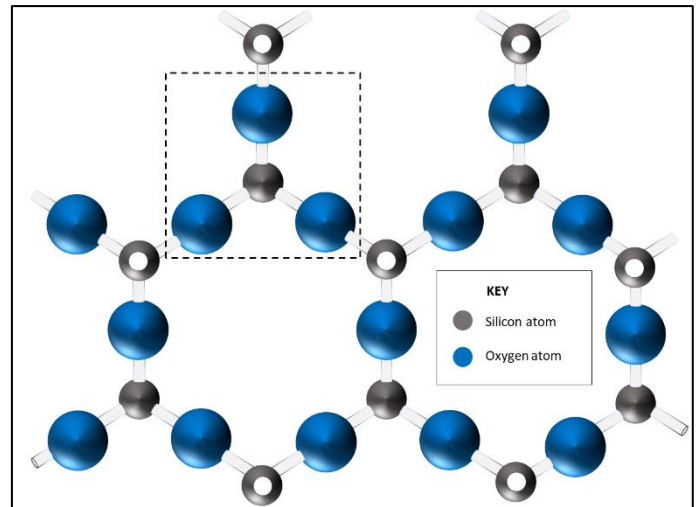


Figure 11: Quartz has an ideal crystal shape as a six-sided prism terminating with six-sided pyramids at each end. Each oxygen atom is shared between two tetrahedra. (Bridges 1997)

1.3.4 Sampling

Proper sampling (box 4) is key to obtain the most accurate and true result. To ensure optimum conditions, the sampling procedure starts before entering the field with preparation. Once at the sampling site, the material should be handled carefully and separated from other material right away to avoid mixing and cross-contamination. Use sealed plastic bags and permanent markers for labelling. All details regarding the sampling method, location and other parameters relevant to the sample material should be noted for later use in a waterproof notebook.

BOX 4

Sample:

A portion of a lot of materials which is taken for testing or for record purposes.

Particle:

A discrete element of material regardless of its size.

(Jillavenkatesa, Dapkunas and Lum 2001)

Note that natural soils are rarely the same from one point in the ground to another. The content and nature of particles varies, but more importantly, so does the arrangement of these. The arrangement and organization of particles and other features within a soil mass is termed its structure or *fabric*. This includes

bedding orientation, stratification, layer thickness, the occurrence of joints and fissures, the occurrence of voids, artefacts, tree roots and nodules, the presence of cementing or bonding agents between particles. Structural features can have a major influence on in situ properties. When retrieving till sediments the sedimentary fabric refers to particles or components that have some sort of orientation that imply the direction of glacial movement.

When returned to the laboratory, the representative sub-sample volume for particle size characterization depends on the expected maximum particle size (figure 12). For small particles sizes, less material is needed. (DGF's laboratoriekomité 2001) Working with material >2 mm requires a larger amount of material to ensure that the sample is representative and the results valid. (Krumbein and Pettijohn 1938)

Maximum particle size [mm]	Sample volume [kg]
>31.5	40
16-31.5	5
8-16	0.7
2-8	0.2
<2	0.1

Figure 12: Sample volume related to maximum particle size of the material.

Plan the laboratory work in detail to make sure there is sufficient time as well as safe and optimal work conditions. Keep the laboratory setting clean at all times to avoid cross-contamination. Be extra careful when sub-sampling, transferring material between containers or when using scales. Each step contributes to the final quality of data and sample loss can be a significant error.

1.3.5 Material variations

At present no standardized method exists for sample preparation of geological material for PSA. At least none that considers different mineral properties and matrices.

In order to eliminate interference and create reproducible data, samples should by default be pretreated when possible. Select a procedure after careful consideration of the content of non-crystalline material:

- Organic material
- Salt
- Carbonates

Depending on the sample origin, it can be necessary to remove organic material beforehand. For soil, oxidation using hydrogen peroxide is standard procedure. Alternatively, when very little material is present, PSA for soil and sediments can be based on an LOI (loss on ignition) sample. Note that high heating temperatures for LOI will affect clay minerals. If the material is heated it will often make the process of suspending the material afterwards a challenge: Once heated the material becomes firm and dispersion is difficult.

For marine samples, it may be necessary to remove traces of salt by rinsing continuously. If salt is not removed, suspended material may be prone to flocculation. The salt minerals can also recrystallize as particles during one of the pretreatment steps (if the material is dried). Marine sediments may also contain biogenic

opal with low specific densities that do not comply with Stoke's law because of their irregular shape. (Buurman, et al. 2001) Such a sample is not suitable for sedimentation analysis.

Samples that are rich in CaCO_3 should be treated with acid to remove the sticky effect caused by carbonates. Not only will carbonates make particles agglomerate, but it will also alter the surface of individual particles and make them appear larger and/or rounder.

Clay minerals tend to form complex colloids with humus. On the surface of the colloids different ions are attached. Because of these colloids, it is likely that the content of silica, iron and aluminum oxides in soil samples can form non-soluble aggregates that affect the PSD or cause flocculation. (Eshel, et al. 2004)

Choosing appropriate pretreatment (and which PSA method to apply in general) can be tricky. They both rely on the sample material (are grams or kilograms available?), composition of material (the presence of organic content, salt, carbonate and type of crystalline minerals), expected size range, timeframe, purpose etc.

All of the common options for pretreatment are described in the next chapter.

2 PRETREATMENT PROCEDURES



2.1 Basic pretreatment

2.1.1 Drying and homogenizing

Pretreatment should prepare the material for analysis. For soil and sediments, particle size is determined on the crystalline material from the original rock material. Not salt, carbonates or organic material that will interfere.

All samples should be dried at $50^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for at least 48 hours, homogenized and split on a rotating sample divider (or similar) to ensure consistent homogenizing. Note that drying at higher temperatures than $50\text{--}60^{\circ}\text{C}$ may alter clay minerals.

Soil samples are crushed mechanically through a 2 mm sieve, using a porcelain dish and mortar (figure 13). A gentle approach is necessary to avoid breaking larger particles into smaller particles. The fraction >2 mm is archived. The fraction <2 mm is used for PSA by LD or sedimentation.

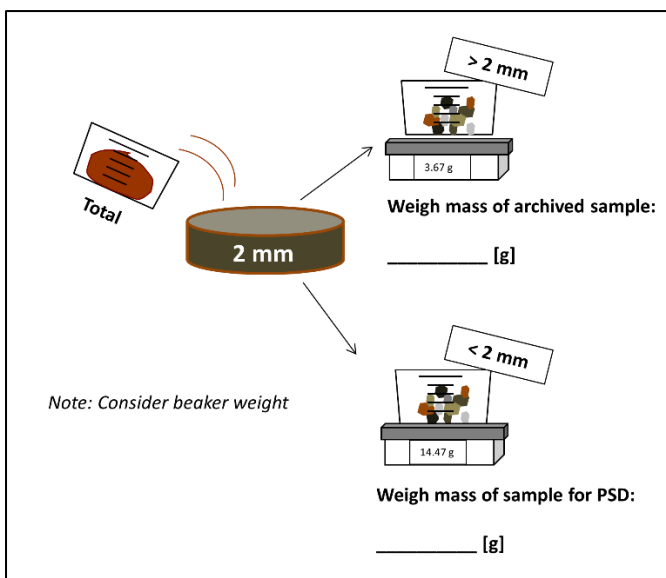


Figure 13: Initial 2 mm sieving of soil prepares two fractions. For sediments 2 mm sieving can be added later.

Note that mass <2 mm can be important for calculations later on.

2.1.2 Salt

Marine samples should be pretreated because they may contain a considerable amount of salt that will hinder dispersion and risk making smaller particles flocculate.

Weigh approximately 50 g of marine sample into a large centrifuge glass (300 mL) and wash with water to remove salt: Rinse the material numerous times with deionized water, centrifuge; remove the water, rinse again, centrifuge and so forth.

2.1.3 Carbonates

The easiest way to remove carbonates in a sample is to treat the material gently with a solution of HCl. Depending on the carbonates present (and the size of sample), start by adding 10 % HCl to a centrifuge glass containing the material. Use a pipette and check the effect: Carbonates decompose into carbon dioxide, water and salt: $2\text{HCl} + \text{CaCO}_3 \rightarrow \text{CO}_2 + \text{H}_2\text{O} + \text{CaCl}_2$.

Rinse the material numerous times with deionized water, centrifuge; remove the water, rinse again, centrifuge and so forth. All excess HCl and salt must be completely removed: If using LD the acid will corrode the flow-cell and inner parts while the salt will have unwanted effects on dispersion, as described earlier.

2.1.4 Finishing basic pretreatment

If preparing for oxidation, weigh the pretreated sample <2 mm into a 1 L heavy-duty Pyrex-type glass beaker of known weight.

Afterwards treat the sample in the individual steps described in the following: Removal of organic material, dispersion, wet sieving, additional dry sieving, and then measurement using the preferred particle sizing technique.

2.2 Organic material, C

2.2.1 Bias caused by humus

Organic material consists of more or less transformed plant material known as humus. Organic material strongly influences the soils ability to retain water and also has an effect on the soil PSD. (Sundberg, et al. 1999)

To avoid bias caused by organic material, it should be removed before PSA for several reasons:

- Organic material will make particles stick together, altering their size.
- Humus are particles and they have a “particle size” too. Overall humus will affect the PSD.
- An inclusion of organic material violates one of the basic principles of LD; that all light passing through the suspension is diffracted. This is not entirely correct for organic material, since the dark material may absorb some light. (Beuselink, et al. 1998)

In conclusion, organic material may heavily disturb the signal from crystalline material during all types of PSA.

2.2.2 Oxidation agent

Oxidation of soil is commonly performed with hydrogen peroxide (H_2O_2) to remove all organic content. H_2O_2 is an excellent oxidation agent since it has moderately strong oxidizing properties and is highly reactive.

At high concentrations H_2O_2 is hazardous and will corrode many materials, including human skin. H_2O_2 may never be in contact with organic solvents (refer to local safety documentation before proceeding) and work is carried out in a fume cabinet using appropriate gloves, safety goggles and suitable laboratory clothing.

Oxidation is carried out by adding H_2O_2 to the sample in a heavy-duty Pyrex-type glass beaker whilst heated. Hereby water and carbon dioxide form: $2H_2O_2 + C \rightarrow 2H_2O + CO_2$.

The temperature may never exceed $60^\circ C$; otherwise, H_2O_2 will boil off as water (H_2O) and oxide (O_2) molecules without reacting with the organic material. For samples that are high in humus content this treatment can take several weeks to complete.

Other oxidation agents may be even more effective in removing organic carbon, i.e. sodium hypochlorite ($NaOCl$) and disodium peroxydisulfate ($Na_2S_2O_8$).

Note that many laboratories choose to oxidize only when the humus content is $>5\%$ (Sørensen and Bülow-Olsen 1994), other laboratories set the limit at $>3\%$. Often sediment samples contain less organic material than soil and oxidation can be disregarded.

2.2.3 Procedure for oxidation using H_2O_2

Weigh approximately 50 g material into a 1 L heavy-duty Pyrex-type glass beaker of known weight. Add 250 mL deionized water and 50 mL 35 % H_2O_2 . Cover the beaker with a watch glass and leave until the following day (figure 14).



Figure 14: Oxidation process after 1 day (left) and from day 2 and onwards (right).

When foaming and fizzling has decreased (approximately 12-24 hours later) the sides of the beaker and the watch glass can be scraped with a rubber spatula. Rinse the loosened material

back into the solution using deionized water. Add 50 mL of concentrated H_2O_2 . Place the beaker on a hot plate (make sure the temperature never exceeds $60^\circ C$) and continue oxidation until all organic material is oxidized: Add 50 mL concentrated H_2O_2 every day (this step usually needs to be repeated 5-6 days or more). Make sure to stir the sample carefully with a glass spatula and check the color of the solution every day.

When oxidation is completed, the sample will turn light brown or yellowish. Never add more H_2O_2 than necessary at a time, high concentrations may cause explosions! On the last day: Raise the temperature to $90^\circ C$ and leave the beaker for evaporation, taking the watch glass off. Make sure to continue to scrape the sides of the beaker with a rubber spatula to make sure as many particles as possible are kept at the base of the beaker.

When the sample is almost dried out, turn off the heat and let it cool before transferring to a heating cabinet and leaving it at $105^\circ C \pm 5^\circ C$ overnight (figure 15).

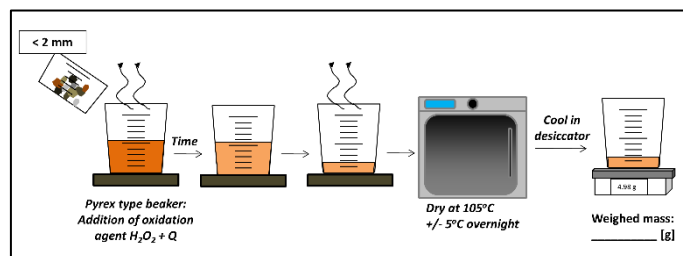


Figure 15: Oxidation of material < 2 mm. Removal of organic material will lighten the color. When oxidation is complete the sample is dried into a powder.

Place the beaker in a desiccator to cool off, then weigh beaker and sample before calculating the loss (= content of organic material). For later calculations, it is important to know the weight of the total (oxidized) sample.

2.2.4 Alternative: Use LOI samples

When working with sediment and especially cores there is not much material at hand to perform many different types of analyses: The procedures must be optimized when using valuable material. If LOI at $550^\circ C$ already has been carried out on a sub-sample (i.e. 2cc), it can be expected that most organic material is removed. The material that is left in the crucible can be used for PSA.

To perform LD measurement subsequently, add droplets of peptizer to the crucible and leave until the following day to dissolve. Then gently stir with a glass spatula and/or a rubber spatula to destroy any clayey aggregates. Once mixed into a paste, this material is measured on LD as-is. Since the LOI material contains the entire particle size range, select an appropriate dispersion system and flow-cell (e.g. 6 mm cuvette).

2.3 Dispersion

2.3.1 Water

Dispersion occurs when objects are distributed over a wide area (or in the case of a solution: Over a large volume). Dispersion can be regarded as a fragmentation process. (Bittelli, Campbell and Flury 1999) The purpose of dispersion is to overcome the binding forces between agglomerated particles and make particles measurable as individual and freely moving objects.

Use deionized water for dispersion. It is vital that the conductivity of the water is low. In most laboratories, the water quality is monitored to ensure high quality, however check before proceeding. Considering the combined effect of Van der Waals forces and electrostatic bonds of particles dispersed in water, an unwanted effect is instant flocculation: Water molecules (figure 16) have dipolar properties as described by H. L. Nielsen. (Dalsgaard, Nørnberg and Kristiansen 2000)

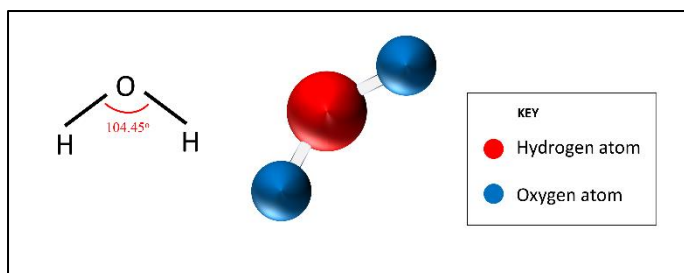


Figure 16: A water molecule has polar covalent bonds between two hydrogen atoms and one oxygen atom.

2.3.2 Agglomerates

If small particles (and especially clay) combine, they form agglomerates that react like a large particle in the suspension and will be measured as such. During sedimentation analysis, these agglomerates will be subjected to Stoke's law at their current size: For all PSA the size of the agglomerate rather than the individual particles will be measured and overall yield a result with a PSD being coarser than it really is.

2.3.3 Flocculation

Flocculation is the process where colloids form out of suspension as flocs or flakes that are larger and heavier than the original, individual particles (figure 17). For PSA flocculation should be avoided (box 5). Flocculation often occurs after mechanical agitation ceases: If stirring or shaking stops, the dispersed clay platelets spontaneously form flocs due to attractions between negative face charges and positive edge charges.

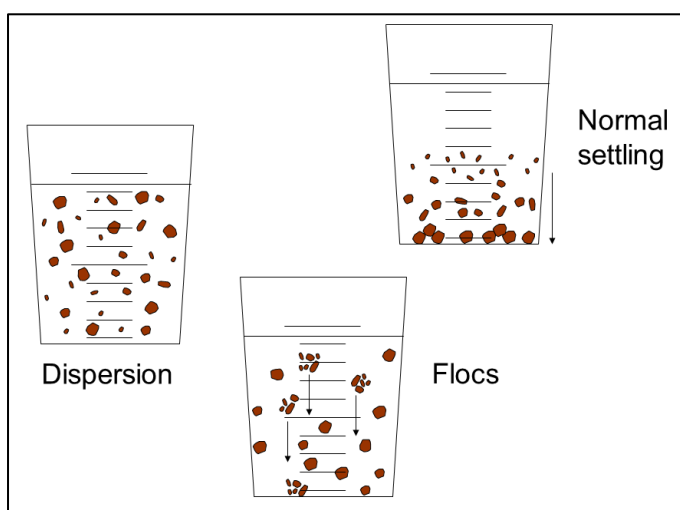


Figure 17: The types of suspensions: Dispersed particles (right after mechanical agitation has stopped), with normal settling and with flocculation.

2.3.4 Exchanging cations

When using a dispersing agent, the Na^+ -ions are exchanged with flocculating ions such as Ca^{2+} and Al^{3+} on the surface of the clay minerals. Clay particles become negatively charged and

thereby repel each other. It is ideal that they split into individual particles as required for PSA.

BOX 5

Flocculation can be avoided in several ways:

If the **concentration** of the dispersed material is kept at a minimum, clay particles will find it difficult to floc. Typically, 20-30 g sample per liter is sufficient and not more than 15 g clay per solution. (Sørensen and Bülow-Olsen 1994) Especially clay minerals with shrink-swell capacities (smectite and vermiculite) may cause flocculation.

Dispersion should take place in a solution of deionized **water** without the normal level of ions present in Danish tap water. If the deionized water is of poor quality (high conductivity level) the surface of the colloids will become charged and the particles will not repel each other well. Thus, larger and more stable agglomerates form.

Dispersing with a **peptizer** can minimize flocculation. By using sodium pyrophosphate, the ions with a high valence exchange for ions of lower valence (Na^+) from the peptizer. The surface now is neutral and there is less risk of flocculation. Adding too much sodium pyrophosphate will have the opposite affect and can produce flocs.

Treating the sample with a reasonable amount of **ultra-sonic treatment** will aid the disintegration of agglomerates. However, too much treatment may break large particles into smaller particles, altering the PSD.

2.3.5 Adhesive forces

Normally, particles <0.0001 mm in water remain continuously in motion due to electrostatic charge (often negative) which causes them to repel each other. Once their electrostatic charge is neutralized, the finer particles start to collide and agglomerate under the influence 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. Van der Waals forces are relatively weak compared to chemical bonds. (Young and Freedman 1996) Attraction increases with higher particle density; thus, the risk of flocculation increases with a higher concentration of particles.

Adhesive forces caused by Van der Waal forces may create colloid particles and induce aggregation of particles which in effect will influence size measurements. Mass forces (gravity, centrifugal forces etc.) dominate >0.050 mm and work against binding forces and the formation of agglomerates. This particularly is enforced if the dispersion is in motion (e.g. stirred).

Electrostatic attraction occurs between particles with oppositely charged surfaces; this can play a significant role on particles <0.050 mm. (Sympatec)

2.3.6 Dispersion agent: Peptizer

Peptizers (box 6) are used as deflocculating agents, i.e. sodium pyrophosphate $\text{Na}_4\text{P}_2\text{O}_7 \cdot (10\text{H}_2\text{O})$ or sodium hexametaphosphate $(\text{NaPO}_3)_6$. Peptizers are salts and soluble in water. Used as dispersing agents they prevent agglomerates from forming and aid the dispersion process. (Krumbein and Pettijohn 1938) The

concentration of the peptizer is important, since a high concentration will turn it into a coagulant and create the opposite effect: Flocculation. (Elonen 1971)

Other studies have concluded that the PSD (especially for clayey samples) is influenced by the dispersion method: The dispersion agent (and its concentration), as well as the use of mechanical end-over-end shakers or ultrasonic sound. (Berthold, et al. 2000)

BOX 6

ALTERNATIVE 1:

Preparation of 1 L basic 0.1 M $\text{Na}_4\text{P}_2\text{O}_7 \cdot (10\text{H}_2\text{O})$

Add 800 mL deionized water into a 1 L flask and gently heat on a magnetic stirrer. Weigh $1.0 \text{ L} \times 0.1 \text{ mol/L} \times 446.06 \text{ g/mol} = 44.61 \text{ g}$ (technical quality) peptizer and wash into the flask with deionized water, wait for it to dissolve. Remove from heat, let cool and fill to the mark with deionized water. Store the solution in a 1 L plastic flask.

0.002 M version (for dispersion and/or wet sieving)

Add 40 mL of 0.1 M solution to a 2 L plastic flask and fill to the mark with deionized water.

ALTERNATIVE 2:

Preparation of 5 L basic 7.5 % $(\text{NaPO}_3)_6$

Add 800 mL deionized water into a 1 L beaker and gently heat on a magnetic stirrer. Weigh 75.00 (technical quality) peptizer and wash into the beaker with deionized water, wait for it to dissolve. Transfer to a 5 L plastic container. Repeat 5 times (in total 375.00 g) and fill to the mark with deionized water.

1.5 % version (for dispersion and/or wet sieving)

Add 1 L of 7.5 % solution to a 5 L plastic container and fill to the mark with deionized water.

Shake all solutions thoroughly before use.

2.3.7 Dispersion of soil samples

Disperse (figure 18) by adding peptizer to the beaker (use measuring glasses of 250 mL and 50 mL respectively): Add 250 mL directly; pour 50 mL into a small spray flask and use this for rinsing of the spatula and ultrasonic equipment later. Carefully destroy any clay with the rubber spatula by scraping against the side of the beaker.

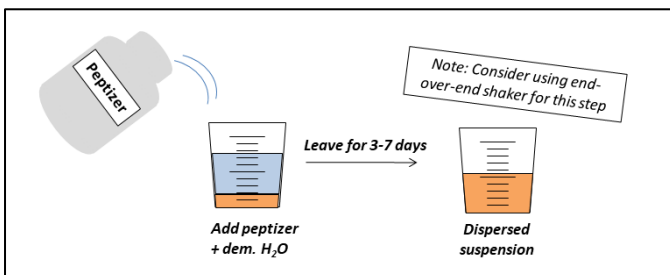


Figure 18: Dispersion using water and adding peptizer.

Treat the sample for 2 minutes with an ultrasonic probe to destroy any possible aggregates. Leave until the following day, covered with a watch glass (and no longer because of possible algae

growth). Check for flocculation (figure 19). If necessary, proceed to neutralize flocculation.



Figure 19: A flask containing material $<0.063 \text{ mm}$ with visible flocs.

2.3.8 Neutralizing flocculation

The signs of flocculation are a clear liquid on top with a jelly-like layer above the sediment. If the glass is tipped the surface of the jellylike layer will stay horizontal.

First, remove the clear liquid, and then add 300 mL 0.1 M $\text{Na}_4\text{P}_2\text{O}_7 \cdot (10\text{H}_2\text{O})$ and stir. Leave until the following day. The sample should be centrifuged (to remove $\text{Na}_4\text{P}_2\text{O}_7 \cdot (10\text{H}_2\text{O})$) and 500 mL 0.002 M $\text{Na}_4\text{P}_2\text{O}_7 \cdot (10\text{H}_2\text{O})$ is added once more. Apply ultrasonic sound for 2 minutes and then leave it 24 hours. Check for flocculation again and continue the treatment until the sample is dispersed correctly.

2.4 Fractionizing

2.4.1 Splitting into fractions

A major part of prepping for LD analysis is ensuring that no interferences affect the measurements. It is therefore desirable to separate a sample into several fractions. These fractions are measured individually and data merged later to obtain a full PSD. When considering available time, sample volume etc. splitting of the material is sometimes left out, or alternative versions are considered.

What is the reasoning behind fractionizing? When preparing the sample for LD analysis it is important to consider adhesive forces and physical properties of different particles:

- Broadly distributed material contains small and large particles which will interact if together in the same suspension.
- Since fine material is not suitable for dry measurements (fine particles may become electrically charged) clay and silt must be measured in a solution.
- Large particles may be difficult to keep in motion in a liquid and are better dispersed using gravitational force.

For these and practical reasons it is best to split the material into at least two fractions, separating fine from coarse particles.

Traditionally, fine particles are separated by wet sieving and coarse particles are separated by dry sieving. Once the sample is dispersed correctly, it is easy to split the finest particles from the rest of the material by wet sieving at a select size (usually at 0.063 mm but 0.038 mm has also been seen). Use peptizer or deionized water in a spray flask.

2.4.2 Wet sieving

The meshes on the sieve should be wet before starting; otherwise, they will initially repel the liquid and material is stopped by the meshes. Stir the sample with a rubber spatula and wet sieve at 0.063 mm (figure 20) with deionized water in a spray flask (or alternative: A solution of peptizer). Use a funnel and wash into a 2 L plastic flask. Ensure that wet sieving is sufficient by checking the color of the water passing through the meshes against a white background (a porcelain bowl can be used): The process is completed when the water is clear. To avoid algae growth, store the flask in a cool place until use.

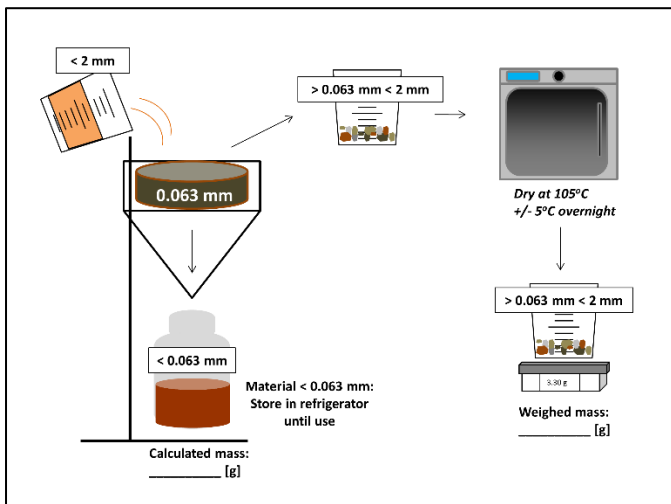


Figure 20: Wet sieving at 0.063 mm.

Wash the remaining particles on the sieve meshes into a porcelain bowl and rinse 2-3 times with deionized water. Transfer using deionized water into a previously dried ($105^\circ\text{C} \pm 5^\circ\text{C}$) and weighed beaker. Carefully remove some of the excess water. Dry the beaker with the material overnight in a heating cabinet at $105^\circ\text{C} \pm 5^\circ\text{C}$. Cool off before weighing the next day. Subtract the weight of the beaker and calculate the mass of the coarsest fraction. From this mass the mass of the finest fraction in the flask is calculated, using the total mass of the sample before wet sieving was commenced.

2.4.3 Dry sieving

If using an LD system with gravitational dispersion, additional dry sieving is called for to be able to measure the coarsest particles in the most optimum way. Choose a reasonable particle size (e.g. 0.250 mm) to split the fraction $>0.063\text{ mm}$ into two sub-fractions.

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 especially during LD data evaluations.

2.4.4 Procedure for dry splitting

Sieve the dry fraction $>0.063\text{ mm}$ using the selected mesh size (figure 21) in a Ro-Tap sieving machine for 20 minutes (refer to the next chapter describing sieving techniques in general).

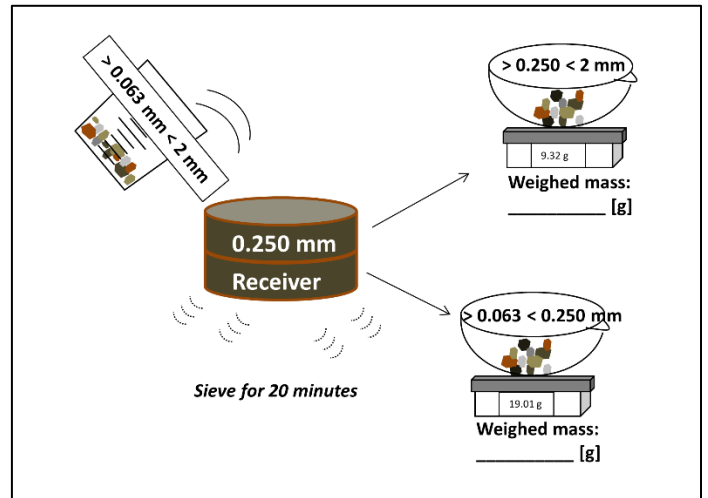


Figure 21: Dry sieving at 0.250 mm produces two additional fractions.

After sieving is completed, empty the meshes carefully using a fine brush (figure 22). Brush the two fractions into two previously dried ($105^\circ\text{C} \pm 5^\circ\text{C}$) and weighed beakers. The mass of each fraction is found directly.



Figure 22: Use appropriate brushes for cleaning sieve meshes: To the left a soft-haired brush for soft (normally fine) mesh material and to the right a wire brush for metal mesh material.

3 SIEVING



3.1 Background

3.1.1 Separating coarse material

The simplest technique to measure the size of an object is by using a measuring tape. This is a one-dimensional method, determining the length of one axis only. If the technique is improved by adding a dimension, the sieve is developed: A two-dimensional method.

Sieving is the process of separating a mixture of particles according to their size by means of one or more surfaces provided with apertures of suitable size and shape while using gravitation. Screen scale is metric and based on a sieve with an opening of 1 mm. (Allman and Lawrence 1972) DS 405.9 describes the standardized Danish sieving procedure.

The use of sieves with different sized meshes is one of the oldest, most reliable, and most commonly used methods for providing PSD. Sieving is widely used for measuring, separating or filtering particles of different sizes: It is simple and relatively low cost to perform. When eliminating person-dependable errors due to weighing and cleaning the meshes, the method is highly accurate and precise on two-dimensional (spherical) materials. When using qualified sieving equipment, the difference between sieves of various fabricate is eliminated.

3.1.2 Sieve

A sieve analysis is carried out in a series of woven-wired square-mesh screens. The screen residue measures the quantity of particles retained (figure 23): It does not describe the size of those retained particles, except that they are *larger* than the screen openings in at least two dimensions. The sizes of the particles that pass through the screen is unknown, except that they are *smaller* than the screen openings in at least two dimensions.

$$\text{Percentage retained} = \left(\frac{\text{Weight of material retained}}{\text{Weight of total material}} \right) \times 100$$

$$\text{Cumulative percentage} = \sum \text{Percentage retained}$$

Figure 23: Calculations of retained material.

Sieves come in different sizes and various materials (i.e. brass, steel etc.). Some sieves are expensive and the finer meshes delicate: Handle them with care. For standard geological sieving analysis, usually a diameter of 20 cm (8-inch) width is used; however, geotechnical sieves can be much wider to be able to handle massive loads of material and/or relatively large particles. For smaller volumes a 10 cm sieve set can be used.

Sieving as means of separation is only possible on coarser material: The mechanical disintegration is not suitable to separate fine particles. Here other methods are necessary (e.g. sedimentation).

3.1.3 Calibrations

The weaving of sieves cannot be perfect. The apertures are not always square, nor are they of exactly uniform size.

Consequently, PSD determined by sieving particles often show a kinky curve when plotted against the nominal sieve apertures. A kink in the size distribution 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 one or more inaccurate nominal sieve apertures cause the kinks. Therefore, a method of calibrating sieves by means of empirical PSDs of hyperbolic shape has been developed. The corrections found successfully adjust the displacement arising from erroneous aperture up to 0.6 mm. (Dalsgaard, Jensen and Sørensen 1991)

3.1.4 Square root and fourth root

Aperture sizes were early on based on a ratio of the square root of two ($\sqrt{2} = 1.4142$) resulting in the aperture width doubling at every second sieve in the series. This was later changed to the fourth root of two ($\sqrt[4]{2} = 1.1892$), where the aperture width doubles at every fourth sieve. These techniques were named square root and fourth root sieving respectively.

Now sieving is usually performed using the halving scale with the following mesh sizes: 2, 1, 0.5, 0.250, 0.125 and 0.063 mm. When working with till sediments the range can be extended to include 16, 8 and 4 mm.

3.1.5 Aspect ratio and shape factor

In geoscience, crystalline particles occur in different shapes and sizes. Two geometrical parameters are used to describe particles: Roundness (sharpness regardless of shape) and sphericity (the degree to which the shape approaches that of a sphere). Three axes can describe the dimensions of any given form: Length, breadth and thickness (or long, intermediate and short), providing four different classes of forms (figure 24). (Zingg 1935)

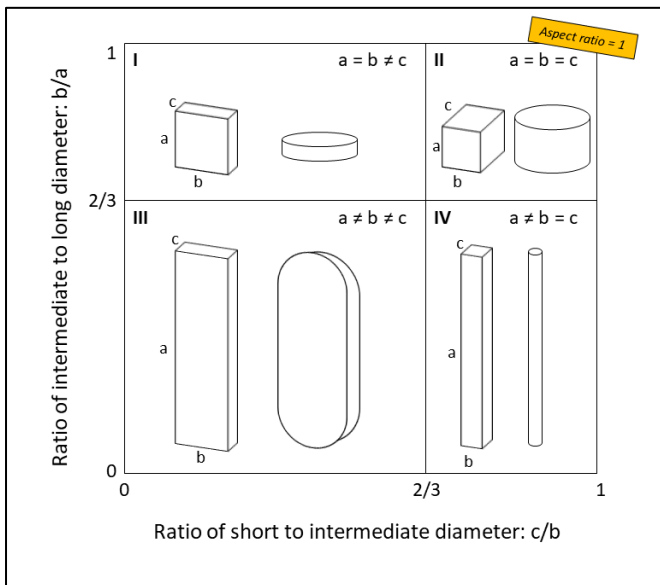


Figure 24: The four classes of particle shape based on ratios of the long (a), intermediate (b) and short (c) diameters. Class I: Oblate (tabular or disc shaped), II: Equant (cubic or spherical), III: Bladed and IV: Prolate (rod-shaped). (Zingg 1935)

The ratio between the shortest and longest axis on a particle is the aspect ratio. When these two axes are equal in length, the aspect ratio is 1. Particles that have high aspect ratios are by this definition not spherical. Chapter 6 deals with this in detail.

Measurements with sieves are depending on the sieve meshes, which yield the two smallest axes on the particle passing through. Meshes are usually square. Particles can therefore be measured smaller than their actual size, since some particles may have two short and one long axis (figure 25).

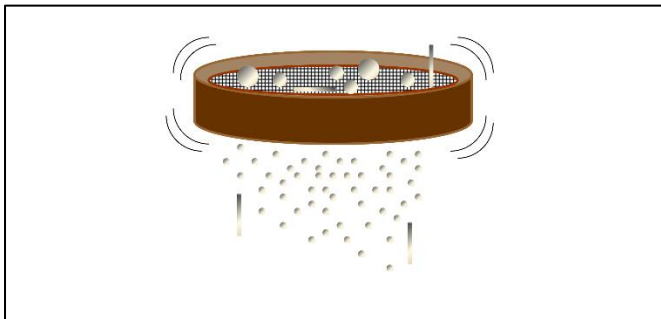


Figure 25: The form dependencies during sieving where some larger particles pass.

This common problem (that non-spherical particles can erect during the sieve procedure and pass with their smaller diameter through the meshes) will alter the PSD: Compared with spheres of the same volume, the sieve information shifts towards a finer PSD. (Sanetra 2004)

There is a certain probability that a given particle will present itself at the mesh opening. The rejection or passing of the particle through the mesh is determined by various factors: The load on the sieve, the particle surface, the dimension and form of the particle and the method of shaking the sieve. Whether or not the particle will actually pass the mesh is dependent on the particle's three dimensions and angle at which it is trying to pass. (Allen 1974)

The sieving process can be controlled by the operator (time, volume of material and means of shaking) but particle properties cannot.

3.2 Measurement

3.2.1 Wet sieving

Fine particles that are dry may choke the meshes and are not properly free-flowing. To conduct a sieving analysis, finer particles are usually removed beforehand by wet sieving. The limiting size for splitting fine from coarse material has been described as 0.075 mm (Allen 1974) and 0.105 mm (Griffiths 1967), therefore it is reasonable to use 0.063 mm to ensure proper separation.

3.2.2 Considerations

Samples may have been pretreated (oxidation etc. are optional, wet sieving is mandatory) and must be dried at $105^{\circ}\text{C} \pm 5^{\circ}\text{C}$. In order to avoid hygroscopic water influencing the sieving loss, the samples are allowed to equilibrate their water content with atmospheric air before sieving.

The initial total (dry) weight is needed before proceeding. Refer to appendix B for a flow chart of all steps of the sieving analysis.

3.2.3 Procedure for sieving

Arrange the series of sieves in a descending order of decreasing particle size. Insert a receiver pan at the base (to collect fine material) and a lid at the top (to prevent escaping particles).

The set is sieved on a sieve shaker or rotational/tapping (Ro-Tap) machine for a fixed time, typically 20 minutes. During this time, all particles must have contact with the meshes so no more than 100-150 g should be sieved at a time. (Griffiths 1967) Longer duration can cause coarser particles to disintegrate into smaller fragments from the mechanical action performed by the sieve.

After sieving is completed each fraction is weighed and the meshes are emptied carefully and without force onto black glossy paper using a soft hair brush (for fine, fabric meshes) and brass brush (for coarse, metal meshes) (figure 26). For larger meshes a needle or other pointy utensil is used. >2 mm should be completely clear of material at all times. Finally, the sieves can be cleaned using pressurized air.



Figure 26: Use appropriate accessories to clean each sieve by hand (left), ranging from brushes of different material and sizes to various needles (right).

The sieving loss should be monitored to ensure quality of the data and track possible sources of errors (meshes can tear, or if sieves are not firmly secured in the shaker particles can escape): Sieving loss should be $< 0.1\%$ for 30 g material or $< 3\%$ for the smallest material of 0.05-0.1 g (Dalsgaard, Jensen and Sørensen 1991) Note that sieving loss can be positive or negative.

3.2.4 Reliability of the method

Sieving by hand is easy to learn but difficult to perform in detail. Results are somewhat subjective to the user and the fabricate of the sieves and sieve shakers. Standard sieving machines should perform a three-dimensional movement: A vertical vibration and horizontal movement (known as "Ro-Tap" for rotating and tapping motion, figure 27). This will jolt the particles at all possible angles, giving them opportunity to align and fall through the meshes. (Thorum 2012)



Figure 27: Sieving should be performed with a certified set of calibrated sieves, and by use of a sieving machine. At the left is the standard Pascall Inclyno model used at the Department of Geoscience. At the right is a custom-built version, suspended from the ceiling (seen at VIA University College in Horsens).

The two principle parameters of length and mass are directly traceable to international standards: A sieve can be calibrated using microscopy, and a scale can be calibrated using reference standard weights.

The sieving time depends on a variety of factors, such as the characteristics of the material, sieve diameter, volume of the charge, relative humidity, and so on, although the rule is that with one additional minute of sieving, if the amount retained on any one sieve changes $< 1\%$, the endpoint of the PSA has been reached.

3.3 Producing data

3.3.1 PSD curves

Data for sieving analysis is normally based on > 0.063 mm only.

Each sieve fraction has traditionally been weighed and the data plotted into a histogram. The PSD can be produced by either a frequency or cumulative diagram, usually both are needed for evaluation.

3.3.2 Software

The Windows software KORN (latest version 1.107) is used at the Department of Geoscience, Aarhus University, for interpreting data from sedimentation and/or sieving analysis. The software was developed from the previous DOS version by Mads Østerby Jespersen during the SESAM project (2001-2010). The KORN software (figure 28) offers various particle size parameters as well as the common PSD diagrams.

Sieving data in KORN can be combined with data obtained by the Andreasen pipette. Alternatively (if KORN or similar is not available), refer to section 5.3 for combining sieving data with data obtained by sedimentation or LD methods in a simple spreadsheet.

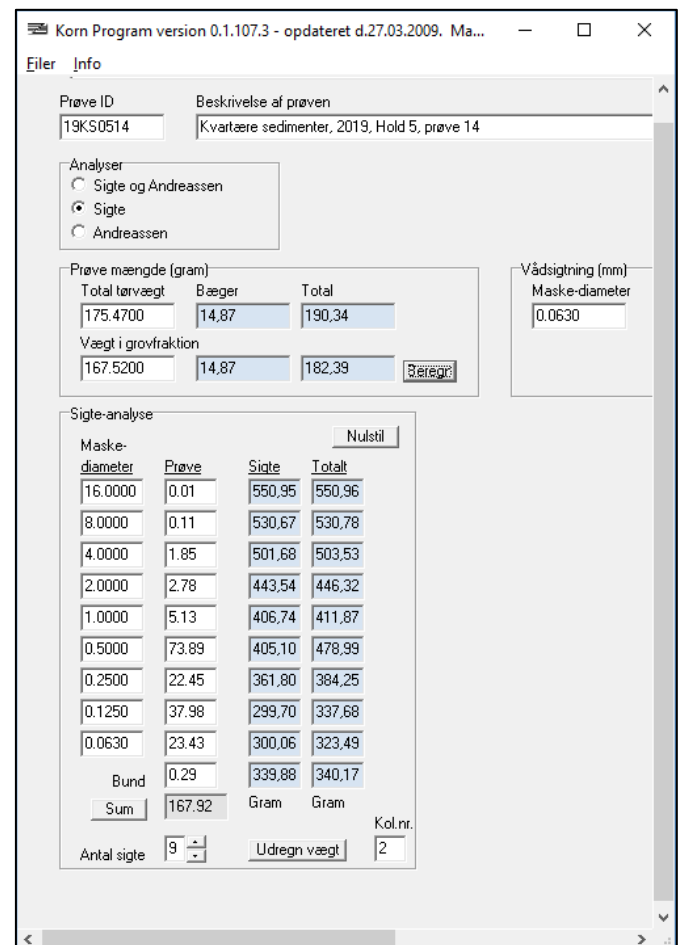


Figure 28: The user interface of KORN, developed by Mads Østerby Jespersen for the SESAM project.

4 LASER DIFFRACTION



4.1 Background

4.1.1 Use of laser diffraction

Laser diffraction or LD (also known as Static Light Scattering) is one of the most widely used techniques today for PSD. LD is described in the international standard ISO 13320.

Experiments as far back as the 1840s resulted in the Fraunhofer diffraction physics, named after the German optician Joseph von Fraunhofer (1787-1826). Fraunhofer assumed that particles are opaque (absorb light completely) and refract the light at low angles. For very fine particles, the Fraunhofer theory is an approximation. Fraunhofer theory assumes that light waves are parallel.

The Danish mathematician and physicist Ludvig V. Lorenz (1829-1891) developed the theory of refraction (figure 29). He published his research in 1890 to a limited audience: "*Lysbevægelsen i og uden for en af plane Lysbølger belyst Kugle*".

The German physicist Gustav Mie (1869-1957) could rediscover the model in 1908. It is now known by the name of Lorenz-Mie theory or Mie scattering. Gustav Mie studied chemistry, mathematics, physics, geology and mineralogy. One of his most prominent contributions to natural science is still used in meteorology today: Determining water content in the atmosphere by radar.

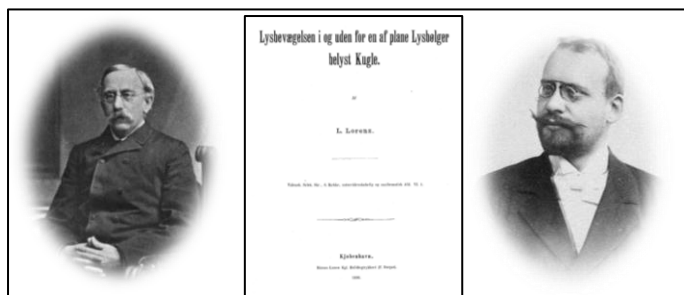


Figure 29: Ludvig Valentin Lorenz (left) and in the middle the front page of Lorenz' famous paper. To the right Gustav Mie (ca. 1905) who rediscovered Lorenz' theory.

Later LD theory was described in the 1980s and innovative companies started to produce high-end instruments. LD instruments

have been developed since then and usage has been further refined during the past decades. LD is used extensively in many industries and research institutions: It is now one of the most important types of analytical tools available. LD is used in material science to describe properties in particle shape, PSD and PSA.

Along with microscopy and X-ray diffraction, LD is one of the most common methods for particle characterization. (Jones 2003) LD instruments are fast and easy to operate: They carry a high reproducibility and mode of detail. Some LD setups are capable of determining particle size up to 8.5 mm i.e. the Sympatec HELOS system that with the correct configuration covers a range up to 8.75 mm. (Sympatec)

4.1.2 Fraunhofer approximation and Mie theory

The diffraction of the laser light results from the interaction of the light with spherical particles and (as mentioned earlier) this can be described mathematically by the Fraunhofer or Mie models.

The term Mie theory is misleading since it is considered to be a solution for the Maxwell equation for the scattering of electromagnetic radiation and not a theory in its own right. The Mie model applies to spheres with diameters between 0 and infinity. Mie evaluation is applicable for monocrystalline materials with a known refraction index (RI) and a particle size <0.010 mm. (Sympatec) When using the HELOS Windox software, re-evaluations based on Mie theory is only applicable up to a focal distance of 200 mm (R4). (Sanetra 2004)

Because different LD systems use different models to evaluate data, data from different systems may not be easily comparable.

4.1.3 Light and particle interaction

LD for PSA is based on the principle, that particles of a given size diffract light with a given angle, the angle being inversely proportional to particle size: A large angle thus indicates a small particle size. (Pedersen 2003)

During the interaction of light with particles, different light phenomena occur at the same time: Diffraction, refraction, absorption and reflection (figure 30). (Rasmussen and Dalsgaard 2010)

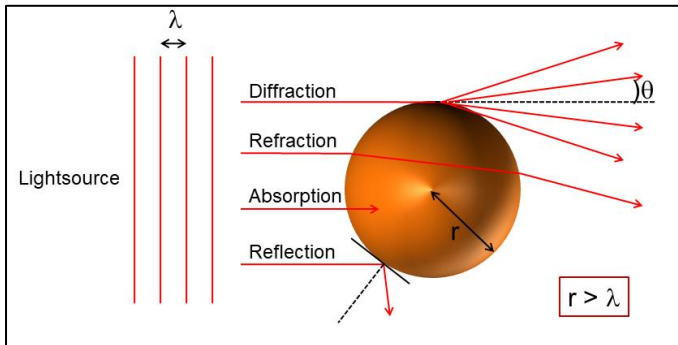


Figure 30: Particle interacting with light waves.

What is the relationship between diffraction patterns and particle size? 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 onto a detector, which records the angular distribution of light intensity $I(\theta)$ (figure 31). (McCave and Syvitski 1991)

$$I(\theta) = \frac{1}{\theta} \int_0^{\infty} r^2 n(r) J_1^2(kr\theta) dr$$

Figure 31: The angular distribution of light intensity. θ is the scattering angle, r is particle radius, $n(r)$ is the size distribution function, J_1 is the Bessel function of the first kind, $k=2\pi/\lambda$ and λ is the wavelength of monochromatic light used by the system.

Having measured $I(\theta)$ the expression above can be inverted to obtain the size distribution. (Singer, et al. 1988)

4.1.4 Diffraction patterns

Either Mie theory or Fraunhofer approximation are used to interpret the scattered pattern and convert it into a size distribution. The acquisition of diffracted light is usually performed with the help of a multi-element photo-detector.

For a single spherical particle, the diffraction pattern shows a typical ring structure (figure 32). The distance r_0 of the first minimum to the center is depending on the particle size. On large particles the incident light will be diffracted in a small scattering angle and on small particles the light will be diffracted in a large scattering angle (figure 33).

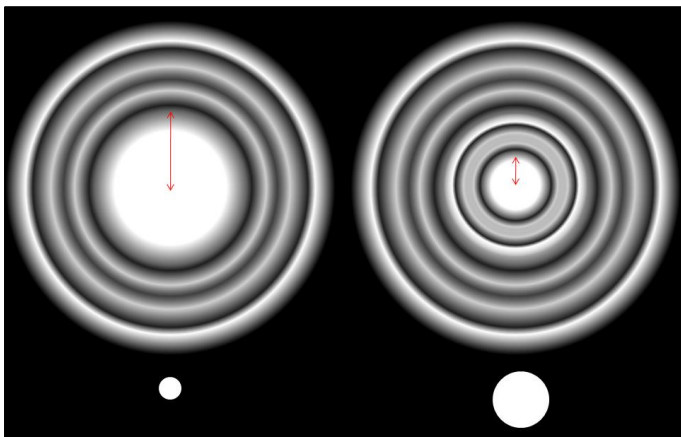


Figure 32: Example of diffraction patterns for spherical particles, small and large. The distance from the center to the first ring is defined as r_0 . (After Sympatec)

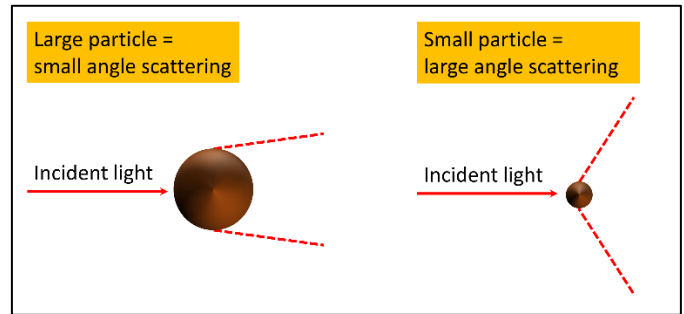


Figure 33: Small and large angle scattering.

The beam intensity (figure 34) is a measure of the volume of the fraction, or how many particles of the same size is present during measurement.

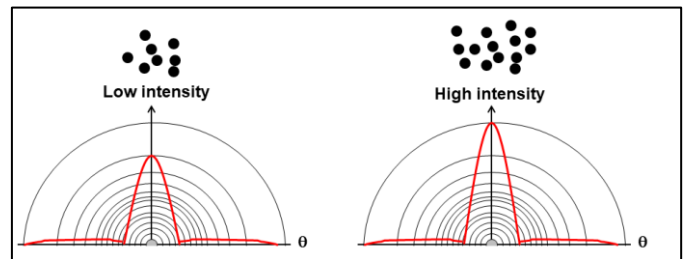


Figure 34: Example of intensity measurement on one particle size. (After Sympatec)

It is assumed that particles are presented to the incident light in all possible directions. This may not always be true: Particles with a large aspect ratio tend to have a preferred orientation. (International Organization for Standardization 2020)

4.1.5 HELOS setup

The HELOS instrument at the Department of Geoscience is equipped with two main modules (figure 35): A unit for wet samples called QUIXEL, containing a reservoir with deionized water. The reservoir is directly connected to a flow-cell with two opposed windows that allow the laser-beam to penetrate the solution. Secondly a shaft unit for dry samples called GRADIS (gravitational dispersion). The latter has a vibrational feeder attached above called VIBRI that enables particles to fall in a controlled manner downwards through the shaft and through the measuring area. Particles must be free flowing and dry to successfully use GRADIS.



Figure 35: Different parts of the HELOS instrument, from left to right: VIBRI module (located above the GRADIS shaft), the QUIXEL module and the 2 mm flow-cell (which is inserted in the measuring area inside the QUIXEL module).

Once entered into the measuring area, light waves target each particle. The diffraction of light is subsequently measured on the far side of the laser unit where a detector with 31 ring elements is located (figure 36). In-between the laser source and the detector appropriate lenses and optics are located.

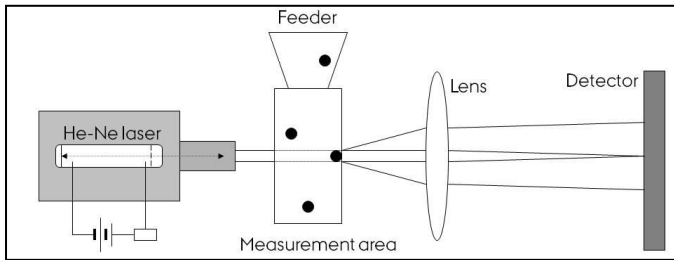


Figure 36: Principle of the Sympatec HELOS system.

4.1.6 Laser source

The instrument uses a helium-neon (He-Ne) laser of protection class/type 34/IP40. The laser beam is created by a mixture of helium and neon gas inside a quartz tube. The beam is formed by photons which are reflected between mirrors on each end of the tube and at the same time stimulating excited atoms to emit photons. The excited atoms appear when the laser medium is connected to electrical power and this process starts spontaneous emission. The process is re-enforced between the mirrors while a certain percentage of the photons is allowed to pass at one end, allowing the He-Ne laser to emit red light at a wavelength of exactly 632.8 nm. (Elvekjær and Nielsen 1993)

He-Ne lasers need warm-up time (>30 minutes) for the system to become stable. The laser beam must not be interrupted during measurement since the evaluation of diffraction patterns depend on a laser-beam always being perpendicular to the lens and detector. The beam's direction and intensity can be influenced by parameters such as touching of the instrument or table surface, closing of door/window and nearby electronic noise.

4.1.7 Detector

The detector is the most sophisticated element of an LD instrument. It has a number of discrete sensitive elements of precise geometry, usually rings (180°) of homogeneous sensitivity and a reflectivity of zero. Detection of very coarse particles require very small elements and an exact alignment of the highly sensitive central-beam (figure 37). (Witt and Röthele 1995)

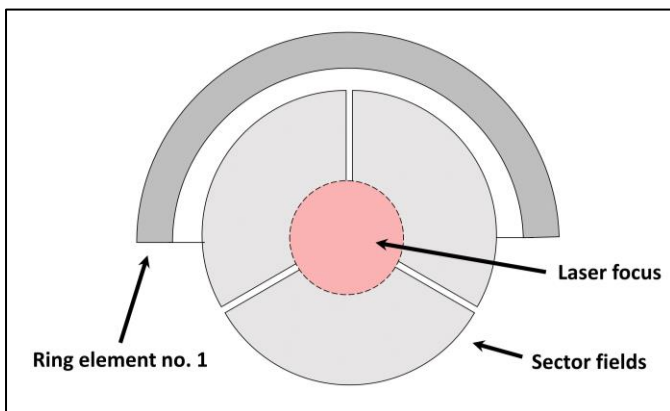


Figure 37: Sector fields in the center are surrounded by ring elements.

4.1.8 Lens systems

The HELOS instrument is equipped with three lenses. The department chose a setup with R1, R4 and R7 respectively. Each lens size is a factor 10 larger than the previous (figure 38). For a particular measurement, a lens is chosen that covers the range of the expected PSD of the material measured. Whenever a lens is chosen, the system will autofocus and align the laser beam parallel to the lens revolver and focus the laser onto the detector.

LENS SYSTEM	Measuring size [μm] of lens		
	R1	R4	R7
Ring element			
(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
Range limit	0.18-35.0	1.8-350	18.0-3500

Figure 38: The three measuring lenses available in this HELOS system: R1, R4 and R7. Sizes measured by ring element no. 1 are considered detection limits. (After Sympatec)

The origin of the PSD is (according to the lens) found at the location where the measurement is 0%. This means that all measurements acquire their first measured data in ring element no. 2.

It is a common misconception that the available size classes of a LD system are directly related to the number of rings in the detector. That is not the case. The measured sizes are to be considered the raw data from which any size class can be deduced. If anything, a large number of rings is the basis of better accuracy of each measurement. Size classes can normally be defined in any way needed; the software simply interpolates the relevant size if they are not already measured directly by the detector.

4.1.9 Flow-cell

The case of the flow-cell (often referred to as the cuvette) is made of hardened steel. It comes in different sizes, depending on the particle size of the material fed into the system.

This HELOS system has two optional flow-cells, a 2 mm and a 6 mm flow-cell (figure 39) allowing for up to 2 mm or 6 mm particles to pass on their longest axis. The 2 mm flow-cell should be used on samples that are split into fractions and definitely <2 mm. The 6 mm flow-cell is used for samples that for one reason or another can't be split into fractions.



Figure 39: The 6 mm flow-cell.

The flow-cell windows are made of quartz which is one of the hardest materials available: The use of quartz minimizes scratches on the glass surface by the particles and ensures the flow-cell a long life.

Any flow-cell needs regular cleaning (refer to the manual for specific procedure with special cleaning tissue) and at times the inner rubber ring must be replaced. During long standby, algae may form. Each flow-cell can be dismantled and opened to clean the inside with acetone and deionized water to avoid particles or algae to stick to the glass. The outside is carefully cleaned with pressurized air and lens paper. Never leave the flow-cell dry: Always keep it filled with deionized water by filling the reservoir to the top, or filling the flow-cell manually with deionized water when kept in storage.

Note that a QUIXEL setup with the reservoir and the 6 mm flow-cell has issues of keeping coarse particles in suspension. When using the 6 mm flow-cell the software automatically raises the stirring speed in the reservoir to avoid this error, but sorting within the flow-cell may occur anyway. An immediate effect of higher pump speed is the formation of unwanted air bubbles, notably the longer a sample is held in the reservoir. These bubbles tend to be led towards the flow-cell where they can't easily escape. Here they affect the measurement by falsifying the result by creating artificial fractions. (Vendelboe 2004-2005) The speed of circulation through the flow-cell can at very high velocities cause particles to orient parallel (rod-shaped particles) or perpendicular (plate-shaped particles) to the direction of flow, causing the measurement to be faulty. (Blott, Croft, et al. 2004)

In conclusion, even though an option, the 6 mm flow-cell setup is not suitable for heavy and coarse crystalline material >2 mm. Generally, inappropriate pumping velocity may lead to sedimentation of the larger particles in the pumping circuit.

4.1.10 Reservoir and sonication

This QUIXEL reservoir has two levels: First filling adds 250 mL deionized water and the second filling adds up to 1000 mL respectively. The suspension is constantly stirred to avoid sedimentation and thus sorting of the particles by size.

Some studies recommend ultrasonic sound treatment up to 2 minutes. Longer treatment times can cause disintegration of some type of particles. (Blott, Croft, et al. 2004)

4.1.11 In-line, On-line and At-line analyzers

The HELOS instrument at the Department of Geoscience is a so-called *At-line* (off-line) analyzer. For industrial purposes, where it is necessary to know particle sizes and material composition of e.g. cement, metal powders or pharmaceutical products during

and after production, PSA can be incorporated directly into the production line (with an In-line analyzer), or a sub-section of it (by line). This secondary line often leads to the quality control line or R&D section where an On-line analyzer performs the PSA (figure 40).

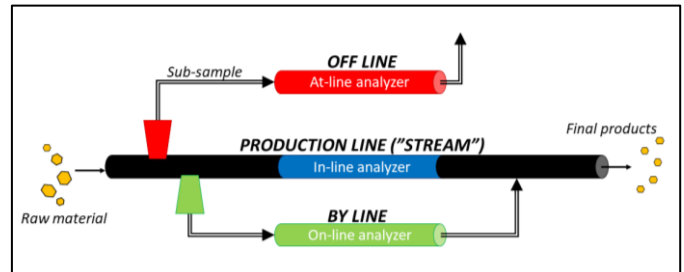


Figure 40: Different types of analyzers.

At-line and On-line analyzers tap into the production line. For industrial production the aim is to interfere with the production line as little as possible while having real-time measurements. This can be obtained by using In-line analyzers. Hereby the material can be continuously monitored by PSA and sampling bias is prevented. In-line measurement is not always an option in a specific industrial setting, then an On-line analyzer (located on the by line) can be used. The material is here returned to the production line after analysis is completed. Note that it is important that the re-circulation line design prevents cross-contamination when returning the material to the production line. In-line and On-line analyses both permit continuous product and process control. A third option is removing a portion (a representative sub-sample) from the main line and measuring sample properties elsewhere on a standalone system. This will inevitably be more time-consuming and it may not be cost-efficient to remove a sub-sample too often. (Heydenrych 2020)

4.1.12 Forced stability issues

It is possible to switch off one or more ring elements. This is carried out by applying Forced Stability (FS) in the HELOS software.

When using R7, the first ring element corresponds to particle sizes >2.46 mm. When selecting FS = 1 (FS1) the first ring element (closest to the center of the detector) is neglected and the instrument does not consider the coarsest particle class. Because of the long focal length, the inner ring element can easily be hit by the laser beam or false light and detect an erroneous signal. It can be necessary to eliminate this part of the signal, when feeding the system with material pre-sieved at 2 mm and seeing a PSD that is coarser than expected. (Romann 2004)

4.1.13 Variance

LD systems are based on first principles, though with idealized particle properties. Thus, calibration by a user is not required. Sympatec informs that the variance on LD is 1.5 % for coarse particles and 1.0 % for fine particles.

Sympatec states that working on 10^7 particles provides a "very low" standard deviation of 1 % on wide distributions. The reproducibility is "very good" as long as the procedures recommended by Sympatec is followed. (Röthele and Puckhaber 2000) As standard procedure Sympatec split at 0.063 mm to avoid interferences. It thus follows that for measurements involving an un-split sample (<2 mm or even <6 mm) the standard deviation will be larger. Therefore at least one split (into two fractions) is preferred, two splits (into three fractions) are optional.

4.2 Measurement

4.2.1 System configuration

There are different options for measurement, refer to appendix C for flow charts of the different approaches.

The instrument as well as software is provided by Sympatec. The operating software (Windex version 5.3.0.0) controls all settings during measurement programs, most importantly:

- Stirring of the reservoir: A suitable pump speed ensures random orientation of particles.
- Ultrasonic sound application: Supports the dispersion of individual particles and prevents air bubbles.
- Vibrational effect for the gravitational feeder.
- Pauses and duration of measurement.

Other parameters are configured to ensure maximum effect and high-quality measurements.

The signal is monitored continuously in the Signal window. Measurement is possible with an optical concentration of 5-50 % but preferably 15-30 %. Too low or too high concentrations yield low-quality results. A minimum of three determinations on the same material (sub-sample or fraction) is carried out, depending on the reproducibility of the measurements: These measurements should be similar, otherwise instrument errors or interferences may have affected the measurement.

Before each measurement, the signal is reset by running a reference (background) measurement on the dispersing agent (a solution of peptizer or air) without particles in the measuring area. If an error occurs, it is an indication that there may be contaminated system units that need to be cleaned manually. Later, the background signal will be subtracted from the detector signal during measurement. The live signals from all detector rings during the blank measurement should be observed: Fluctuations, negative values or overload readings may indicate faulty ring elements. (International Organization for Standardization 2014)

4.2.2 Choosing appropriate lens

As an example, for a sample pre-sieved at 0.25 mm one would logically expect to use the R4 lens on the Sympatec HELOS system for the fine fraction: R4 covers <0.35 mm and provides many relevant raw data points. However, due to software and sieving issues of geological material it has proven necessary to use R7 to ensure no coarse material is cut off. The R7 lens covers <3.5 mm and unfortunately provides fewer relevant data points.

4.2.3 Standard procedure for wet measurements

As mentioned earlier, due to complex mineralogy and wide size range, soil samples require fractionation and separated treatment of fines and coarse material. (Sympatec 2002)

If the sample has been split into several fractions <2 mm, the 2 mm flow-cell is used. If the entire sample is run as-is (or the operator is inexperienced or unsure of the maximum particle size), the 6 mm flow-cell is used.

The software ensures that ultrasonic sound treatment is applied at the beginning of each measurement program (60 seconds) in order to degas the solution and disperse particles. This is followed

by a short break (10 seconds) before the measurement (20 seconds).

Before the addition of material to the reservoir, the signal is reset with a reference (blank) measurement: Add 10 mL of peptizer to the reservoir. Different wet measurement options using QUIXEL are seen in figure 41 and described in detail below.

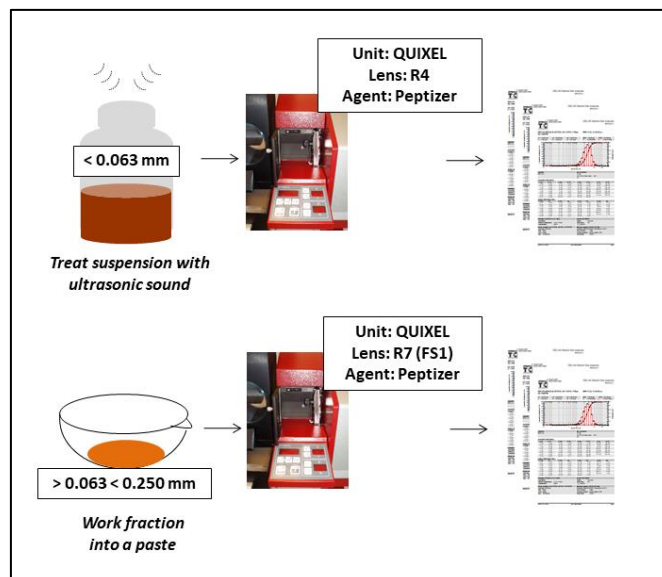


Figure 41: Example of two alternative measurements of wet material: Finest particles in a suspension and coarser particles in a paste.

<0.063 mm measured with R4, 2 mm flow-cell

The fraction in the flask is treated 2 minutes with an ultrasonic sound probe. The sample (in a plastic flask with a stopper) should be shaken well before use. At first 20 mL is added to the reservoir with a small plastic beaker that fits within the flask mount and large tweezers (quickly submerged in the plastic flask). Observe the concentration. Each sub-sample is measured at least three times. After completing all analyses, the sub-sample is discarded and the remaining material in the flask can be stored cold.

If the optical concentration is too low a second sub-sample can be added. If the concentration of the suspension is still very low and there is little or no material left, it is optional to run with a low-filling of the reservoir to artificially increase the concentration.

0.063-0.250 mm measured with R7 (FS1), 2 mm flow-cell

The fraction in a beaker is worked into a homogenous paste right before the analysis, using a few drops of deionized water and a glass spatula. Then a sub-sample (2-4 scoops with a spatula, observe the concentration) is added to the reservoir. Each sub-sample is measured at least three times. After completing all analyses, the sub-sample is discarded and the remaining sample can be air dried and archived.

If too much liquid is added to the paste, the material will be sorted in the beaker and it will not be possible to sub-sample representative material. It is better to leave the sample until the following day (to let water naturally evaporate), then re-do the mixing again.

Alternative: Use the 6 mm flow-cell on an unsplit sample. Settings for QUIXEL is R7 (FS1) and the sample is worked into a homogenous paste. Measure three times as described above.

4.2.4 Standard procedure for dry measurements

During cold and dry periods of the year, it may be necessary to moist the air in the laboratory while measuring dry material. Otherwise there is a risk of particles being subjected to static electricity.

The coarse fraction will be underestimated if too few particles are being measured. Therefore, Sympatec has developed a gravitational dispersion system (GRADIS): The software controls the flow of particles through the shaft by a certain feed rate (here: 50) created by gentle vibration from a second unit (VIBRI). This will ensure a continuous flow of particles into the measuring area. It is better to opt for a slow feed rate, otherwise particles will block each other and the diffracted light will contain too much noise.

0.250-2 mm measured with R7 (FS1)

A reference is run (on air) between each measurement. A suitable amount of material is placed in the funnel and fed down through the shaft during measurement (figure 42). After the measurement, the remaining parts of the material can be brushed down the shaft and collected in the drawer at the base of the unit.

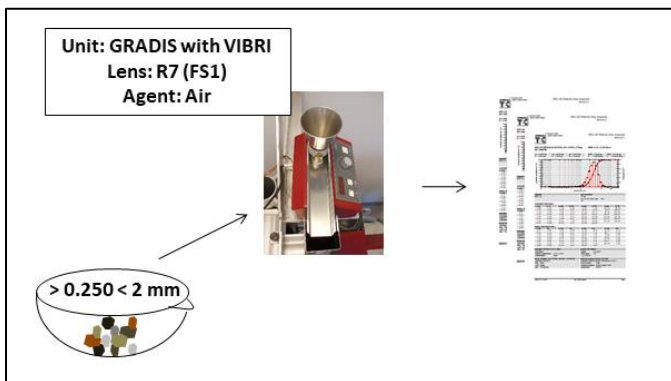


Figure 42: Example of measuring dry fractions.

The funnel and drawer are cleaned with pressurized air and/or a vacuum cleaner. Dry measurements are non-destructive so the entire fraction can be archived and re-used for other purposes if desired. However, expect to lose some particles in the cleaning process.

4.2.5 Contamination

The contamination index is based on an absolute reference measurement which is carried out right after thorough cleaning of all parts of the system and stored in the database. The contamination index is also dependent on the intensity of each detector ring element.

In the QUIXEL unit the reservoir and flow-cell are rinsed in-between measurements with deionized water until the contamination index is <1 %.

Wide distributions require little contamination to affect the PSD, narrow distributions are less of a problem. Also note that the calculation of the contamination index requires that the dispersing medium during normal measurement is the same as during the absolute reference measurement.

4.2.6 Reliability of the method

The Windox software ensures that the material in the QUIXEL unit is mixed while under constant stirring and treated with ultrasonic sound to de-gas the solution and prevent clay aggregates to

form. The length of time of applying ultrasonic sound in the reservoir should be considered: It should never exceed 60 seconds, since crystalline particles (i.e. mica) can break when affected. (Elonen 1971)

The optical concentration (C_{opt}) is the obscuration of the center of the detector caused by particles in the laser beam (figure 43). I_{ref} is the mean intensity on the detector center during reference measurement and I_{meas} is the mean intensity on the detector center during a normal measurement.

$$C_{opt} = (I_{ref} - I_{meas})/I_{ref}$$

Figure 43: Optical concentration.

An optical concentration in the reservoir of approximately 15-30 % is preferred, according to Sympatec the optimal concentration is 30-40 % but varies with the material.

It is desirable with an acceptable signal-to-noise ratio with respect to precision: 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, the light scattered from a particle may be scattered again by a second particle (known as a multi-scatter effect), causing errors. (Bittelli, Campbell and Flury 1999)

Note that obscuration is the light that does not reach the detector. During measurement a fraction of the transmitted light is blocked (and scattered) by particles: Obscuration = 1 - transmission. In other words, 100 % transmission is the laser intensity with no particles present (obscuration = 0 %). When adding particles to the system, the obscuration increases and the transmission decreases. (Horiba Instruments 2010)

Although small amounts of material are necessary to run a LD measurement it is a precondition that the selected material is homogenous and representative of the entire sample, in order for the results to be easily reproduced. (Møller and Pedersen)

4.3 Producing data

4.3.1 Software

Raw data (figure 44) is stored in a database and is non-changeable. Readouts however are always changeable: Whenever it is necessary to test different settings on an existing measurement, the measurement can be loaded into the software and the data manipulated by changing the settings as desired.

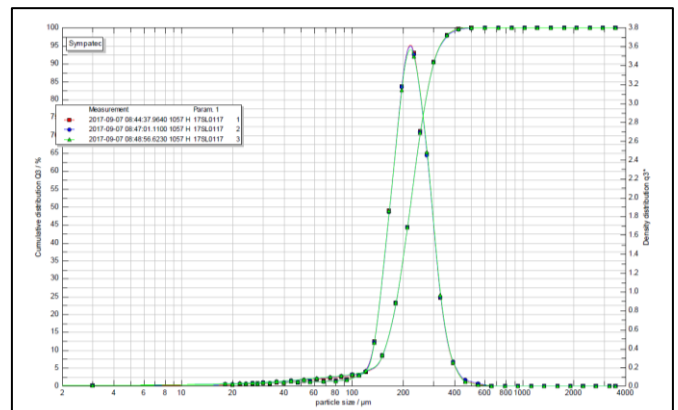


Figure 44: Example of data generated by Windox. Note the high reproducibility: The three measurements are hard to distinguish on the diagram.

All measurements produce a data report based on a personal template. The report by default includes many standard particle size parameters (box 7).

BOX 7

SMD and VMD:

Terms such as Sauter mean diameter (SMD, the surface-volume mean), and Volume mean diameter (VMD) are sometimes used in PSA. SMD is defined as the diameter of a sphere that has the same volume-to-surface ratio as a particle of interest. VMD refers to the midpoint particle size (or mean): Here 50 % of the volume of the particles is smaller, and 50 % of the volume of the particles is larger than the VMD.

Frequencies (in the HELOS software denoted $p\beta$) and cumulated diagrams are used to present PSD. Where data has been provided within a particle size range, the end point is used for computations of (x, y) . When creating histograms, the midpoint of the interval is typically used for (x, y) .

The Q(x) evaluating software by Sympatec furthermore provides a third output option to express the distribution density by volume: A smoothing of the frequency values, $q\beta^*$ (figure 45).

$$q^*(x) = \frac{\Delta Q(x_i)}{\Delta \ln(x_i)}$$

Figure 45: Equation for smoothing the frequencies, $q\beta^*$ in the software.

4.3.2 Re-evaluation using Mie theory

For a normal measurement the HELOS uses the Fraunhofer approximation (in the Windox software denoted *HRLD*) by default.

Data can be re-calculated using the Windox software. Mie theory can only be applied on measurements carried out using R4 (or smaller) lens and then it only makes sense on finely grinded, homogenous minerals with a known refractive index. (Sanetra 2004) There are certain difficulties to consider when applying Mie theory to soil and sediment samples so it is not used very often. (Micromeritics Instrument Corporation 2006)

Both Fraunhofer and Mie models can be used for particle sizes >0.010 mm. Mie theory provides the most accurate results <0.010 mm since this model adds the effect of the absorption of light as well as the refraction of light through the particle itself. The use of the Mie theory presumes the precise knowledge of the complex refractive indices of the particle and medium. The application of Mie theory provides good results for fine, highly transmissive, spherical materials with well-known physical properties. The choice of the refractive indices can have a great influence on the calculated results of the PSD. In conclusion, applying Mie theory is not recommended for the analysis of a mixture of components with different refractive indices such as geological material. (Sympatec)

4.3.3 Export

All raw data must first be imported from the database into the Q(x) software. To keep track of the measurements it is easiest to keep a log (in a spreadsheet) with the unique database timestamp and the operator's notes taken during measurement.

From each set of measurements, the PSD is evaluated and a representative measurement is chosen.

Using a mean PSD of the three measurements proves to be disadvantageous in the software later, since certain information linked to the raw data is lost.

Q(x) provides solutions for merging the PSDs found by measurement from different lenses, as well as calculating statistical values and create user-defined tables for the output. Also, Q(x) has an option to import external data (i.e. retrieved by traditional sieving) to combine with present LD data and thereby create a total PSD based on several different analytical methods.

4.3.4 Merging: Automatically (default)

When two or more measurements have to be combined into one single PSD curve the merging can be carried out automatically by the Q(x) software (figure 46). Merging is carried out by selecting the measurements in the database, manually typing in each fraction's mass, choosing the fractions that are to be combined and then merging them into one PSD. Then a specific output class is defined. Select an output type which is a combination of the raw data measured by the lenses (R4+R7), as well as the clay-silt-sand information when those are applicable. The selected output is exported to a text-file that can be imported to a spreadsheet. With the acquired raw data the operator is free to interpolate new particle sizes or calculate preferred parameters.

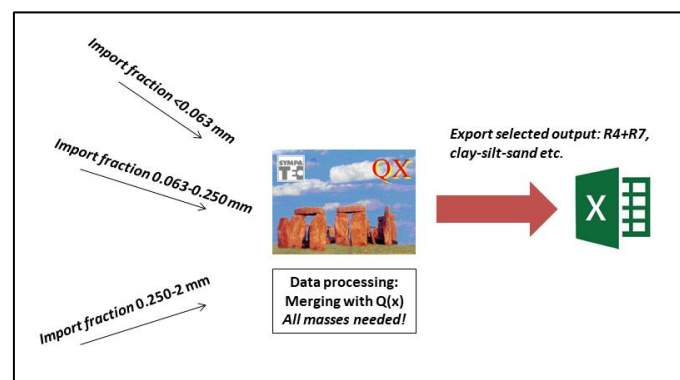


Figure 46: Merging all measurements into a full PSD.

How is default merging carried out? The Q(x) software uses a mathematical approximation to fit the curve-gradients of two distributions in the "best" coordinate. This to make sure that the curves are indeed overlapping. However, the best particle size suggested by the software can be near the limit of the measuring range of the lens, where the curve from R4 is breaking off. If so, the PSD is not optimum and therefore it is not always considered the most realistic choice for a suit of samples to merge by default.

4.3.5 Merging: Linked Analysis Special

A work-around to receive more measuring points without splitting the sample, is to run a representative sample twice, each time choosing a different lens (e.g. first R4 and later R7). Afterwards the measurements can be merged (or linked) in a single coordinate: Using this special function the software takes the particle size range of each lens into account. Therefore, it is a precondition that the ranges overlap in at least one coordinate.

There are mathematical disadvantages when using Linked Analysis Special, i.e. the software does not allow for the individual masses of each fraction to be typed in and thus does not take

the mass of each fraction in relation to the entire sample into account. (Sanetra 2004) Another disadvantage is that the necessary material volume for the QUIXEL unit is twice as big as normal: Effectively this procedure resembles the measurements of two samples. For the GRADIS unit the material can be reused. Sadly, recycling material is not an option on this HELOS system: On newer models it is possible to switch lenses once material is in the reservoir of the QUIXEL.

The evaluation of the ability to connect in a single coordinate uses a criterion, which is calculated from the gradient of the two PSDs to the right and left of the connecting particle size value. A smooth connection (figure 47) is achieved if the gradient of the transformed PSD in the fine range coincides with the gradient of the PSD in the coarse range. (Sympatec)

$$K(x) = - \left| \log \left\{ \left[\frac{qF(x)}{qG(x)} \right] \cdot \left[\frac{QG(x)}{QF(x)} \right] \right\} \right|$$

Figure 47: Formula calculating the smooth factor $K(x)$.

Low K -values corresponds to the best smoothing. (Sanetra 2004)

Criteria for smooth factor $K(x)$:

$QG(x)$: PSD in the coarse range.

$QF(x)$: PSD in the fine range.

$qG(x)$: Linear density distribution value in the coarse range.

$qF(x)$: Linear density distribution value in the fine range.

It seems a reasonable approach to select a size for linking located at a distance from the limits of the lens such as 0.1 mm (R4). Unfortunately, more often than not it has been difficult to achieve a nice smoothing using this technique.

4.3.6 Comparing data sets

In calculating results based on LD-theory, there are certain aspects to note.

Users often have different preferences and resource limitations: Changes in the applied technique and software can alter the observed and evaluated measurements; without question there is a need for standardization of procedures. (Callesen, Palviainen, et al. 2017) (Borggaard, et al. 2011)

Generally, it can be troublesome and confusing to compare results derived from different types of instruments, since various systems may operate with different hardware configurations and calculation models. (Callesen, Andersen, et al. 2016) Differences in the number of ring elements in the detector, or type of lens system and optics in different systems will affect the PSD. (Fisher, et al. 2017)

It has proven difficult to find equations that accurately describe the relationship between LD results and traditional sieving/sedimentation results. None of the models derived fit into an over-all conclusion. Especially between LD and the Andreasen pipette method there is no common denominator observed: LD measurements are likely to be dependable on the mineral type. To complicate the matter further: Studies have concluded that fitting performance varies with particle size, not only type and shape of particles. (Bah, Kravchuck and Kirchhof 2009)

However, depending on the material composition the PSD can be quite similar. As an example, the sample 1MP-V3 (windblown sand from Staby, Western Jutland) was measured on two different instruments and the two distributions are very similar (figure 48). (Rasmussen and Dalsgaard 2017)

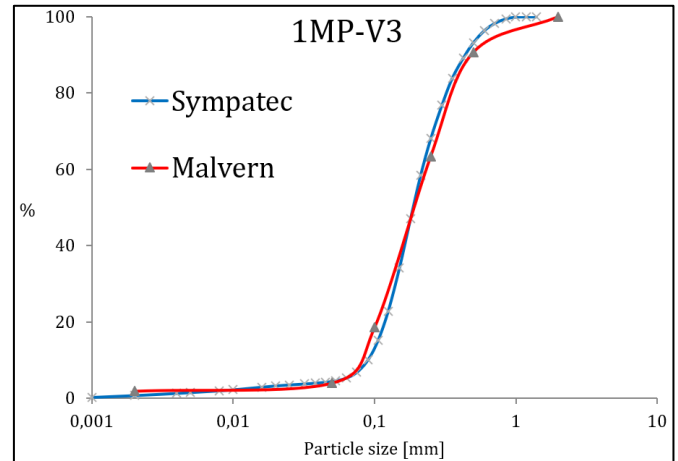


Figure 48: A sample measured on two different instruments: The Malvern Mastersizer E (University of Copenhagen) and the Sympatec HELOS (Aarhus University).

4.3.7 The clay-silt boundary

As mentioned in the first chapter, there are different definitions of the boundary between clay and silt, one has traditionally been at 0.002 mm.

As an example, the content of clay and silt is of high importance for soil treatment procedures within agronomy. For many reasons (economic, ecological, practical) it is essential to determine accurate particle sizes near the clay-silt boundary.

Samples containing clay particles are almost always measured coarser by LD as opposed to data obtained by traditional methods. I.e. particle sizes evaluated by the sedimentation method to be coarse clay is by LD determined as fine silt. For practical use LD experiments indicate that the limit between clay and silt is at 0.006 mm. (Rasmussen and Dalsgaard 2017) Other studies have concluded that clay particles determined by sedimentation are registered on the laser instrument as being in the size range 0.005-0.008 mm because of their platy form. (Konert and Vandenberghe 1997) A recent study support these ideas and observations: The research used SEM to conclude that silt-sized particles were wrongly included in the clay fraction identified by the sieve-pipette method. (Yang, et al. 2019)

4.3.8 A note on specific minerals

Detector signals and distribution diagrams should be evaluated continuously, here are some common observations:

- If the PSD changes rapidly while measuring in the QUIXEL unit, the sample may contain magnetite: When present in a solution, magnetite produces a PSD with a single peak that suddenly changes to a bimodal diagram on the next measurement; the concentration drops and a coarsening effect takes place. This is a sign of the individual magnetite particles agglomerating into larger objects.
- Samples rich in iron may coat the glass of the flow-cell windows, causing a high contamination error and the need to clean more often.
- For podzol B-horizons there may be interferences from iron- and manganese oxides.

5 ANDREASEN SEDIMENTATION



5.1 Background

5.1.1 Settling

Sedimentation occurs when particles settle in a suspension. In theory, particles with the largest diameter will settle faster than particles with smaller diameters.

The pipette sedimentation method was developed by Alfred Herman Munk Andraesen (figure 49) and makes use of a cylinder and a pipette with a constant volume, known as the *Andraesen pipette*. (Andraesen 1939)

The method requires moderately skilled personnel to carry out the analysis. This method is limited to particle sizes <0.050 mm. (Krumbein and Pettijohn 1938)

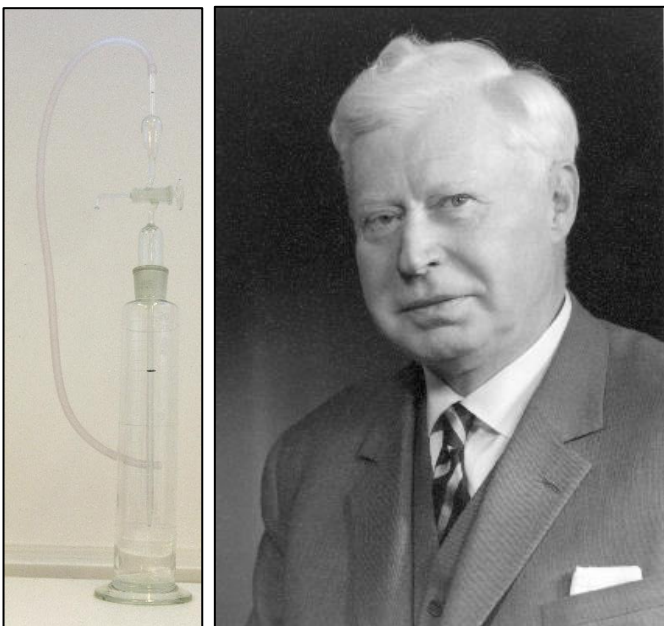


Figure 49: The Andraesen pipette used in the sedimentation analysis is named after its inventor, Alfred H. M. Andraesen, a Danish chemist. (Right photo: Elfelt)

The sedimentation method is based on Stoke's law. The concentration of the particles has to be held at a reasonable level, typically 5-10 g per liter is sufficient. Stoke's law assumes that the suspension has a single particle density. However, soil is not heterogeneous and particles not always spherical in the measured range. (Bah, Kravchuck and Kirchhof 2009)

5.1.2 Stoke's law

When particles with a known density settle in a solution of known volume, the particle size at a certain location in a cylinder can be calculated to a certain point in time. Stoke's law (figure 50) is based on spherical particles with smooth surfaces. A large particle will settle faster than a small particle.

$$v = \frac{h}{t} = \frac{2gr^2(\rho_p - \rho_f)}{9\eta}$$

$$t = \frac{18\eta h}{gr^2(\rho_p - \rho_f)}$$

Figure 50: Stoke's law used for time calculation at an exact particle size.

- v: Velocity of settling particle (cm/s)
- ρ_p : Particle density ("crystalline" material: 2.65 g/cm³)
- ρ_f : Fluid density (1.00 g/cm³)
- g: Gravitational force (981.6 cm/s²)
- η : Fluid viscosity at 20°C (0.01002 poise or g/(cm · s))
- h: Height of fall (cm)
- t: Time in seconds (s)
- r: Particle radius (cm)

The cylinders used are filled to 15 or 20 cm. Assuming that each extract removes exactly 10.00 mL, h is found to be 0.4 cm on these types of Andraesen cylinders. For inexperienced users, some pipettes offer the option of reading additional volumes over 10.00 mL.

5.1.3 Brownian motion

In a fluid, sub-micron particles will move randomly in a process known as Brownian motion. This process is based on thermal energy fluctuations that causes molecules in a suspension to interact and be in constant motion. Some particles collide and are forced into random directions and moved in a process called diffusion.

Diffusion is size-dependent. Brownian motion therefore affects settling of particles during sedimentation: If a number of particles subject to Brownian motion are present in a given medium and there is no preferred direction for the random oscillations, the particles will tend to spread evenly throughout the medium over time. In result, very fine particles may remain indefinitely in suspension and do not settle. Stoke's law assumes that particles for sedimentation analysis are large enough to be unaffected by Brownian motion. (Encyclopedia Britannica 2006)

5.2 Measurement

5.2.1 Sedimentation procedure

Refer to appendix D for a flow chart of the sedimentation analysis. Material is wet-sieved at 0.063 mm.

Since Stoke's law is dependent on temperature: The analysis should be carried out in a climate-controlled laboratory or simply by using a temperature-regulated water bath.

The rate of settling of a given particle size in all cylinders is calculated beforehand using Stoke's law and a fixed time schedule prepared (figure 51). The analysis is performed on three consecutive days. On the first day the cylinders and beakers are prepared, as well as an extract retrieved at $t = 0$ s (0.063 mm). The second day sedimentation is re-started and performed over 10 hours. Beakers with extracts are left to dry overnight. Finally, on the third day, all extracts are weighed and a PSD is created.

Time schedule														
Location	A	B	C	D	E	F	G	H	I	J	K	L	M	N
Time	00:00:00	00:00:45	00:01:30	00:02:15										
	A ₀	B ₀	C ₀	D ₀										
	00:02:32	00:03:17	00:04:02	00:04:47	00:05:45	00:06:30								
	A ₁	B ₁	C ₁	D ₁	E ₁	F ₁								
					00:06:17	00:09:02								
					E ₁	F ₁								
	00:09:50	00:10:35	00:11:20	00:12:05			00:14:30	00:15:15						
	A ₂	B ₂	C ₂	D ₂			G ₁	H ₁						
							00:15:35	00:16:20	00:17:02	00:17:47	00:19:00	00:19:45	00:20:30	
					E ₂	F ₂	G ₁	H ₁	I ₁	J ₁	K ₁			
									00:21:32	00:22:17	00:23:02			
									I ₁	J ₁	K ₁			
									00:24:20	00:25:05	00:25:50	00:26:35	00:27:20	
									O ₁	N ₁	L ₁	J ₁	K ₁	
											00:31:30	00:32:15	00:33:00	
											L ₁	M ₁	N ₁	
											00:34:01	00:34:46	00:35:31	
											L ₁	M ₁	N ₁	
											00:41:18	00:42:03	00:42:48	
											L ₁	M ₁	N ₁	
											00:43:59	00:44:44	00:45:29	00:46:14
											E ₂	F ₂	G ₂	H ₂
											00:52:44	00:53:29	00:54:14	00:54:59
											H ₂	I ₂	J ₂	K ₂
											00:57:14	00:57:59	00:58:44	01:00:34
											I ₂	J ₂	K ₂	L ₂
											01:09:34	01:10:19	01:11:04	
											L ₂	M ₂	N ₂	
	02:28:30	02:29:15	02:30:00	02:30:45	02:34:15	02:35:00	02:43:00	02:43:45	02:47:30	02:48:15	02:49:00	02:59:02	02:59:52	03:00:37
	A ₃	B ₃	C ₃	D ₃	E ₃	F ₃	G ₃	H ₃	I ₃	J ₃	K ₃	L ₃	M ₃	N ₃
	09:36:17	09:37:02	09:37:47	09:38:32	09:42:02	09:42:47	09:50:47	09:51:32	09:55:17	09:56:02	09:58:47	10:03:21	10:04:06	10:04:51
	A ₃	B ₃	C ₃	D ₃	E ₃	F ₃	G ₃	H ₃	I ₃	J ₃	K ₃	L ₃	M ₃	N ₃

Figure 51: Exact time schedule for one person, operating 14 cylinders (A-N). For this example, the cylinders are initially filled to 15 cm and the temperature is 22°C.

Depending on personal experience and available equipment, it is recommended to analyze a series of 7-14 samples at a time. It is a precondition that the sample is dispersed in a solution of deionized water and has been pre-sieved at 0.063 mm. Finally, an appropriate (and known) volume of peptizer should be added to the suspension beforehand to avoid flocculation of the material.

Wash all glassware thoroughly (use a solution of HCl or deconex) and rinse with deionized water. Dry one beaker per extract (0.063, 0.032, 0.016, 0.008, 0.004 and 0.002 mm respectively) at 105°C ±5°C, cool in a desiccator and weigh in advance. Shake the flask containing the dispersed sample well and pour into the Andreasen cylinder. Often fine material is left in the lid or on the sides of the flask: Make sure to rinse all remaining particles into the cylinder before proceeding, use deionized water. Insert the Andreasen pipette and turn the top to a neutral position to let the fluid and air reach equilibrium. Fill carefully to the mark with deionized water. Clip each cylinder firmly into its position in the water bath and turn on the heater.

The next day the samples have reached the temperature used for calculation. Each sedimentation analysis must be started in a way that doesn't allow for any of the extracts to be sampled at the same time. Refer to the time schedule based on the exact laboratory conditions (cylinder volume and temperature) and use a timer all day to be precise (figure 52).



Figure 52: Sedimentation analysis with the Andreasen pipette method.

At the exact calculated times 10.00 mL of the suspension (the "extract") is carefully led up and into the pipette and immediately out into a beaker (figure 53). Quickly thereafter the pipette is rinsed by leading deionized water up into the pipette and out again into the same beaker. Over time, the particle sizes left in suspension become smaller according to Stoke's law.

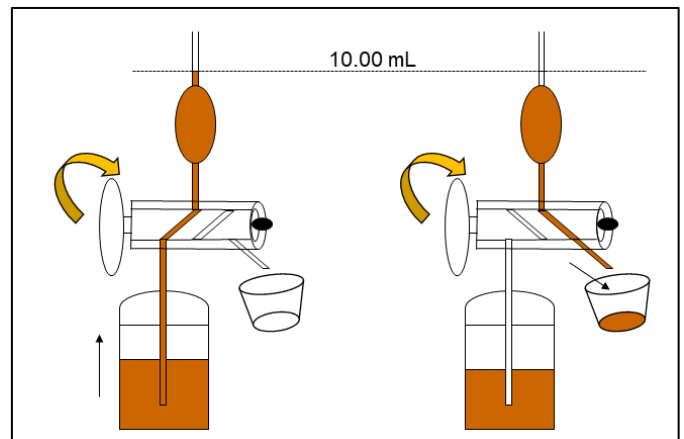


Figure 53: In the Andreasen pipette 10.00 mL sample is led into the pipette bulb (left) and then released into an empty beaker (right).

Dry all beakers at 105°C ±5°C overnight, cool in a desiccator and weigh. The mass of each fraction is determined (corrected for weight of dissolved peptizer) and the PSD can be found.

5.2.2 Reliability of the method

The most stable position of a settling non-spherical particle is the one in which the maximum cross-sectional area is perpendicular to the direction of motion.

This however decreases the settling velocity during sedimentation analysis and the fine fraction is overestimated. (Eshel, et al. 2004) According to this theory, laminar particles (clay) do not settle like a spherical particle during sedimentation. They either cut downwards through the liquid in abrupt cycles with minor

pauses, very much similar to a leaf cutting through air when falling from a tree. Or they can fall slowly like a parachute, their wide area hindering them to reach the expected settling velocity according to their volume. (Berthold, et al. 2000) The result is a finer PSD than expected.

Disturbance of the settling (creating currents and turbulence in the cylinder) must be avoided. Some disturbances are caused by temperature fluctuations giving rise to convection currents in the cylinder. This can be minimized using a temperature-controlled environment and providing sufficient time for the sample in the Andreasen cylinders to reach the same temperature as their surroundings. Sedimentation is also affected if the cylinder is not entirely vertical or when a too fast extraction disturbs the settling near the base of the capillary tube.

The Andreasen pipette is designed to allow a small amount of liquid to be retained in the capillary tube which gives rise to a systematic positive error. For some type of distributions, the error is substantial. To avoid this error the sample residue can be removed before the next extraction by slowly leading it back into the suspension. This procedure however may generate another error: Disturbance of the sedimentation. (Allen 1974) Experienced users may become skilled in letting back the pipette residue without disturbing the settling particles.

The density of the particles in a non-heterogeneous suspension will always be an approximation. Commonly 2.65 g/cm^3 is used as density for soil and sediments originating from parent material like granite and gneiss: These rock types produce particles of mainly quartz (2.65 g/cm^3) and feldspar ($2.55\text{-}2.76 \text{ g/cm}^3$).

Extracting the sample at the exact time (down to seconds) is crucial for the analysis to be of high quality. In figure 54 a series of settling velocities for different particle sizes has been calculated according to Stoke's law. Note that incorrect extraction (removal too late) turns out to have a larger effect on the PSD in the beginning of the analysis (yellow area) rather than when the particle size has increased (green area): Once the slope stabilizes (horizontal), the effect of being late will not change the particle size (or the PSD) significantly.

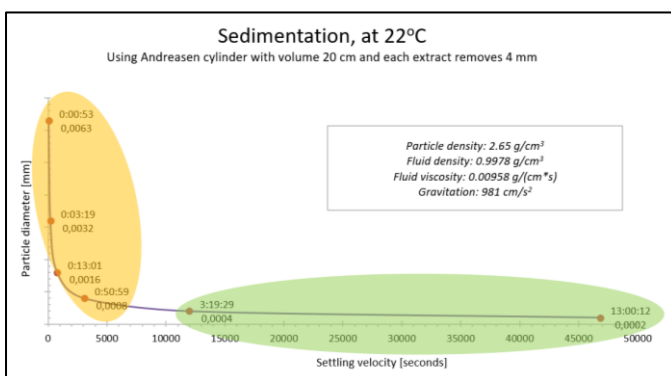


Figure 54: The error when extracting too late becomes smaller over time.

In the example (and according to the calculations using Stoke's law) the extract for 0.032 mm should be retrieved at $t = 3 \text{ min } 19 \text{ s}$ after starting the sedimentation. If by mistake 30 s is added, Stoke's law can be reversed to find that the particle size retrieved is instead 0.030 mm . This error corresponds to a deviation of 6% from the expected particle size value. If the same mistake (30 s late) is repeated for the extract at 0.004 mm , the calculated corresponding particle size is still 0.004 mm and deviation 0% .

Most of the time a PSD can be created based on incorrect extractions. The only requirement is to note the exact time (minutes and seconds) that the operator is late, where after the corresponding particle size can be recalculated.

Andreasen cylinders are all designed for the same purpose but the outcome from the production process by different glass companies has turned out slightly different. Original cylinders and pipettes can be hard to come by nowadays. Wear and tear, especially near the pipette or on the capillary tube, may have required the glass to be mended several times and the intended fixed volume may have changed. Therefore, each cylinder and pipette bulb should initially have their exact volume (mL) determined. This is important in order to know how much peptizer (g) the pipette bulb contains exactly: During data processing the mass of the peptizer should be subtracted from the mass of each extract.

The start volume should be as large as possible: Usually the cylinder can be filled to 20 cm and it is recommended to utilize its maximum capacity. However, in order for the operator to make a realistic schedule that fits a normal work day, the start volume can be reduced to 15 cm or similar, just make sure to recalculate the time schedule accordingly. The reduced height will speed up the process of sedimentation (the particles will reach the base of the cylinder faster), but this will also raise the error margin so it is not a recommended approach.

5.3 Producing data

5.3.1 Visualization

Results are expressed as equivalent spherical diameters based on mass.

For easy data manipulation, use the KORN software or a simple spreadsheet such as Microsoft Excel (figure 55).

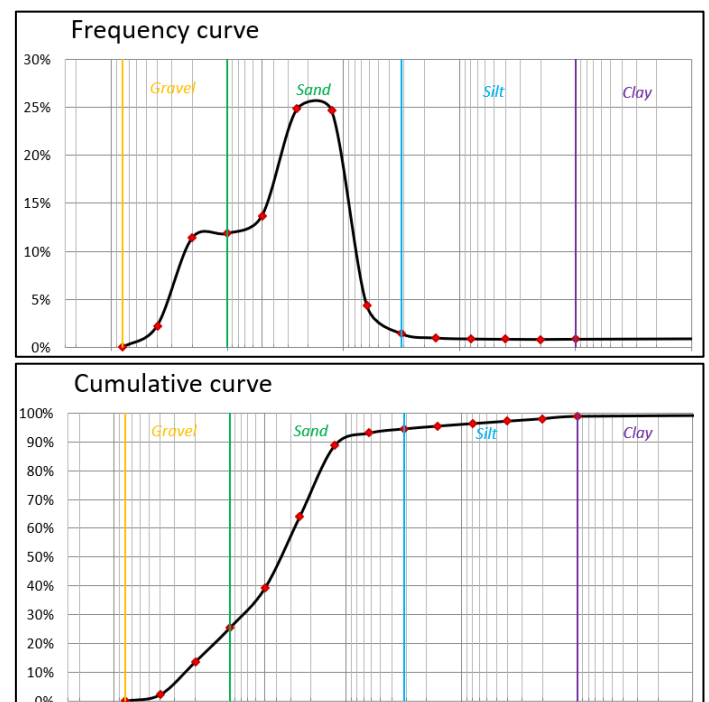


Figure 55: Using a spreadsheet for PSD. In this example data from the sedimentation analysis has been merged with data from the sieving analysis, presenting a total PSD in the range $0.002\text{-}1.6 \text{ mm}$. Frequency (top) and cumulative (below) diagrams.

6 DYNAMIC IMAGE ANALYSES



6.1 Background

6.1.1 Shape and form

Manual (static) microscopy is now replaced in many laboratories with automated (dynamic) image analysis. Choosing image analysis is often driven by the desire to generate results that are accurate, sensitive to second populations, contains shape information and includes image representations of the particles.

Different terms (including shape, morphology and form) have been used by different authors to describe the external geometrical expression of sediment particles and rock fragments. (Blott and Pye 2008)

Note that shape is two-dimensional or “flat” (an area that is defined by a line) and form is three-dimensional or “fat” (forms have height, width and depth) (figure 56).

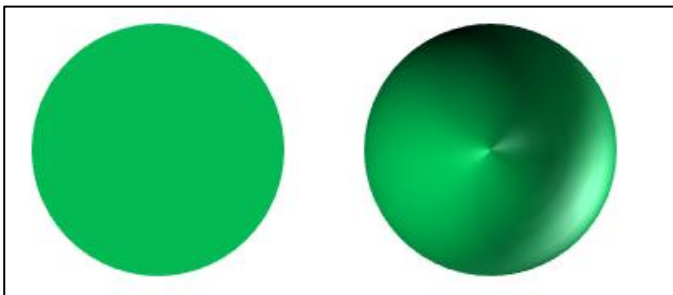


Figure 56: Shape (two-dimensional) and form (three-dimensional).

In a transport medium, particle shape will affect transportation and final settling of a particle. Collision and force during transport determines how fast a particle will become more rounded. Wind rather than water will make particles more spherical.

Particles with irregular shapes will be deposited with a preferred orientation. Therefore, shape often affects size measurements.

Shape and form are key to understanding sediment attributes like compaction, deformation resistance, flow characteristics and particle density. These properties are often vital to understand and control in geotechnical engineering, especially within construction.

6.1.2 Particle shapes

Shape of particles supports determining the properties of soil and sediments: It influences the strength and stability of the material. The shape of particles varies from very angular to well-rounded (figure 57). Angular particles are generally found near the rock from which they are formed. Angular particles have greater shear strength than rounded ones because it is more difficult to make them slide over one another.

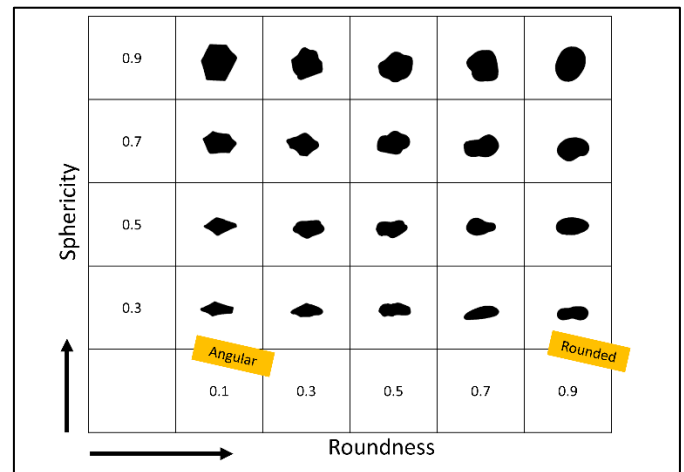


Figure 57: Visual chart for estimating roundness and sphericity of sand particles. (Krumbein and Sloss 1951)

Two general shapes are normally recognized: Bulky and platy.

Cobbles, gravel, sand, and silt particles cover a large range of sizes; however, they are all *bulky* in shape. The term bulky is confined to particles that are relatively large in all three dimensions, as contrasted to platy particles, in which one dimension is small as compared to the other two. The bulky shape has the following four subdivisions (figure 58) listed in descending order of desirability for construction purposes. (Embuido, Azucena and Davis 1991)

- *Angular* particles are those that have been recently broken up and are characterized by jagged projections, sharp ridges and flat surfaces. Angular gravels and sands are gen-

erally the best materials for construction because of their interlocking characteristics. Such particles are seldom found in nature, however, because the weathering process does not generally produce them. Angular material must usually be produced artificially, by crushing.

- *Sub-angular* particles are those that have been weathered to the extent that the sharper points and ridges have been worn off. Glacial sediments are often irregular shapes with round edges (sub-angular and sub-rounded). From glaciers, deposition varies from well-graded basal tills and boulder clays to poorly-graded deposits in moraines and outwash fans.
- *Sub-rounded* particles are those that have been weathered to a further degree than sub-angular particles. They are still somewhat irregular in shape but have no sharp corners and few flat areas. Materials with this shape are frequently found in stream beds. If composed of hard, durable particles, sub-rounded material is adequate for most construction needs.
- *Rounded or well-rounded* particles are those on which all projections have been removed, with few irregularities in shape remaining. The particles resemble spheres and are of varying sizes. Rounded particles are usually worn by water or air and found in or near stream beds or beaches.

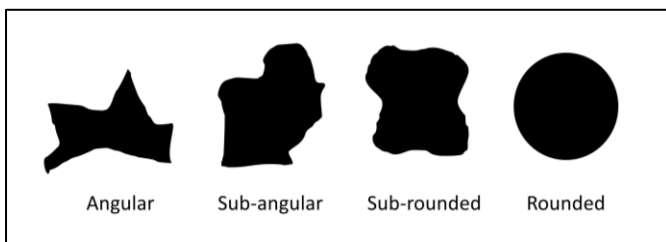


Figure 58: The four bulky shapes.

Platy (or flaky) particles are those that are flat and plate-like. As mentioned earlier, clay and mica are common examples. Because of clay's shape, these flaky particles have a greater contact area for moisture and are normally undesirable for construction purposes.

6.1.3 Instrument basics

The Sympatec QICPIC instrument measures particle size as well as particle shape parameters. This instrument is compatible with the international standards ISO 13322-1 and 13322-2. (International Organization for Standardization 2014)

As with the Sympatec HELOS system for coarse material, the Dynamic Image Analyses (or DIA) system is using gravitational dispersion with a vibrational unit. However, unlike the HELOS system using a red laser and a detector system to convert a diffraction pattern into equivalent particle sizes, the Sympatec QICPIC system uses a green laser pulse at 532 nm and a high-speed CMOS camera to capture black and white images. (Rasmussen and Mootz 2012) The CMOS camera is a simple "active-pixel" sensor based on metal-oxide-semiconductors (MOS) technology.

At the Department of Geoscience, the lens M8 is installed. M8 has a magnification of 2. The size of 1 pixel is 0.010 x 0.010 mm. The physical particle size range covered by the M8 lens is 0.020 mm and up to 20 mm (ISO conformity up to 6.820 mm). However, considering the pixel size, Sympatec suggest the range starting

point at 0.060 or 0.080 mm for proper shape recognition. Windox software (version 5.3.0.0) controls all measurement settings.

6.1.4 Camera

A camera frame rate of 70 Hz (70 fps) is used at the Department of Geoscience. Despite the selected frame rate, the *projected* frame rate is always 20 fps. The resolution of the camera is 1024 x 1024. The technology is limited by the captured resolution of the objects: A minimum of $3 \times 3 = 9$ pixels is necessary to describe a shape and thus provides an acceptable size and shape result. 3 pixels still provide acceptable size results. The uncertainty of particle size increases with very low pixel numbers.

6.1.5 Dispersion system

Moving particles can be introduced into the measuring area in different ways. At the Department of Geoscience particles are fed using a vibrational feeder (VIBRI): Gravitational force then leads the particles through a vertical shaft for dispersion (GRADIS). The feed rate (the velocity of the feeder vibration) can be anywhere from 0 to 100 % but should not exceed 50 %.

The shaft is designed to enhance particle distribution in the measurement area and in front of the camera (figure 59). This will minimize the risk of particles blocking each other on the captured images.

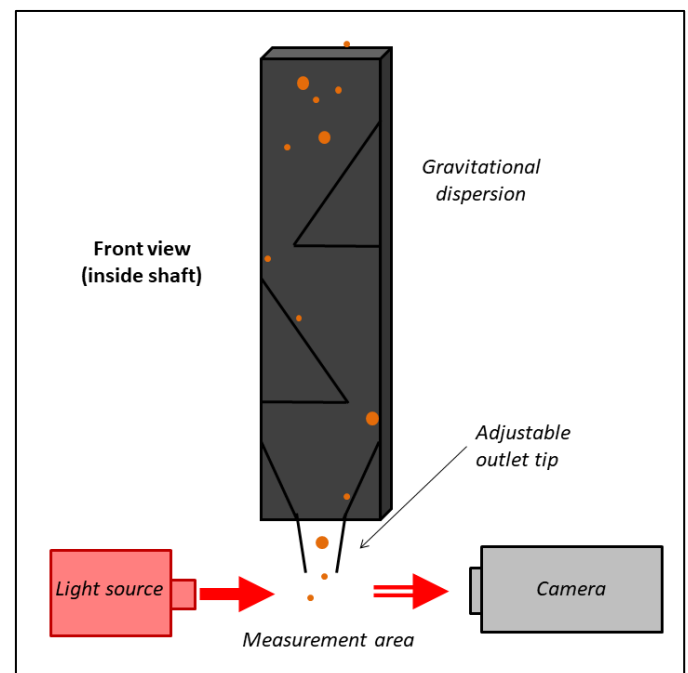


Figure 59: Distribution of particles inside the GRADIS unit.

The GRADIS unit ensures that particles are measured in the center of the measuring area, capturing the full particle and not cutting its edges off. Otherwise size and shape parameters cannot be deduced. To optimize the centering of particles, the end of the shaft consists of an outlet tip of various sizes. Use a tip size of 2 x the expected size of the largest particle.

6.1.6 Size classes

In theory, an infinite number of particle sizes (and classes) can be evaluated. The information is present as long as the database contains images that can be re-evaluated. On a daily basis 20-50 classes can be used for the data report, more than 100 classes are impractical to use. According to Sympatec, if using too wide

a selection, the sensitivity of evaluated results will become poor. Too narrow a selection, the percentages of the measured classes will be small (some even empty), yielding low statistical relevance and oscillating diagrams.

Size classes should be kept constant over a series of measurements to obtain comparable results.

6.1.7 Diameter descriptors

Numerous definitions of size related to shape are used internationally. The Feret diameter and EQPC are both used by the QICPIC software and described below.

Feret diameter: The measure of the size of an object along a specified direction (in general the distance between two parallel lines restricting the object perpendicular to that direction). Maximal or minimal Feret diameter are found after consideration of all possible orientations (0-180°). The Feret diameters for a sufficient number of angles are calculated and their maximum or minimum is selected. $X_{F, \max}$ is the longest distance, $X_{F, \min}$ is the shortest distance. Also, $X_{F, \max 90}$ is the Feret diameter measured at an angle of 90° to $X_{F, \max}$ (figure 60).

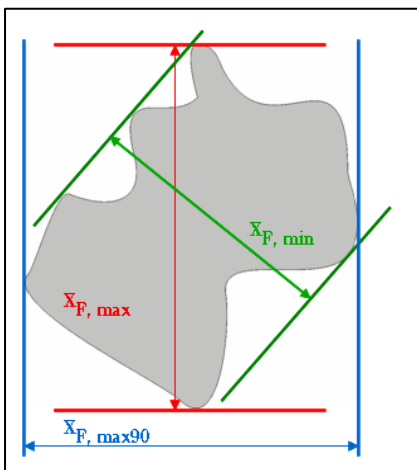


Figure 60: Definition of the Feret diameter. (After Sympatec)

EQPC: EQPC (or Equivalent Projected Circle) is the diameter (X_{EQPC}) of a sphere that has the same area as the projection area of the (maybe irregular) particle (figure 61).



Figure 61: Definition of EQPC. (After Sympatec)

6.2 Measurement

6.2.1 Startup

Refer to appendix E for flow charts describing two versions of DIA. Ensure that the sample (or sub-sample) is homogenous and representative of the original material. The material is preferably wet-sieved beforehand to avoid coating.

Also avoid contamination of the QICPIC system and GRADIS unit by using only coarse material.

Select a suitable measurement program that is optimized for the type of material. Choose class limits depending on the expected particle size range, including buffers.

6.2.2 Feeding

The number of particles to be used is size dependent: Having larger particles, there is less particles at work in the same volume of material, and more particles are needed to ensure a statistically valid measurement (figure 62).

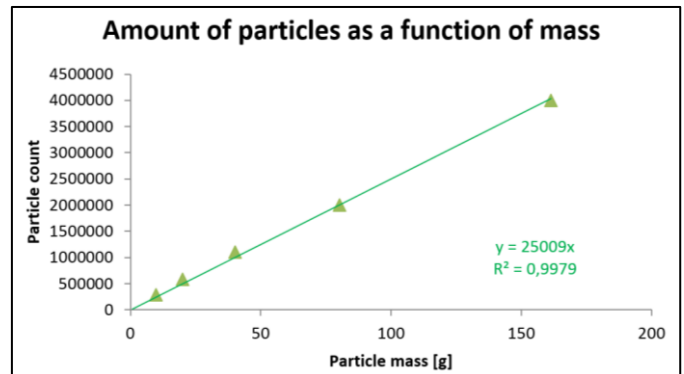


Figure 62: Example of the relationship between the number of particles and mass.

Material is placed as close to the mount of the chute as possible, to avoid time-out errors in the software: Too low concentrations in the measuring area will initiate a cleaning process. The feed rate is 20 %. For normal use, a single measurement of each sample/material is sufficient (figure 63).

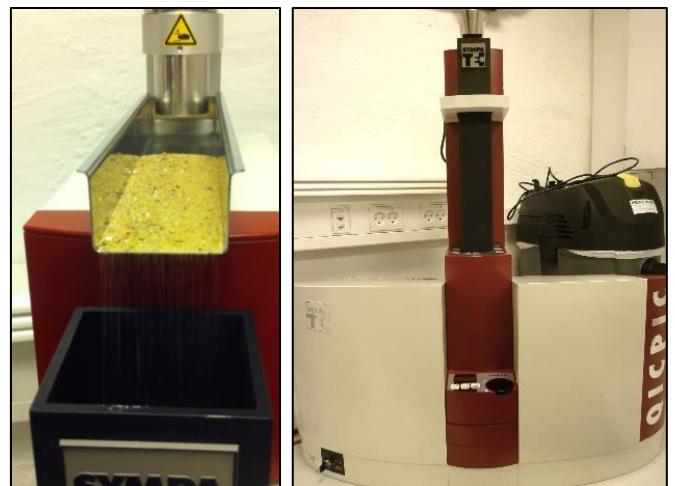


Figure 63: The dispersion unit (a vibrational feeder) to the left, introducing material into the measurement area through the GRADIS. The QICPIC system to the right.

Refer to the online videos for visualization (box 8).

BOX 8

Feeding: <https://youtu.be/5fZVpaButFE>

Captured particles: <https://youtu.be/vjBtGzWDges>

The optical concentration C_{opt} is an indicator for the volume flow of the particles. It is the ratio between the number of black pixels

(particle pixels) with respect to the total number of pixels (figure 64).

$$C_{opt} = n_{black}/n_{total}$$

Figure 64: C_{opt} is the ratio between particle pixels to the total number of pixels.

C_{opt} is preferably 10-20 % but the value depends on many measuring factors (size, feed rate etc.) and thus is rarely within this range.

The number of particles measured is saved in the data report. The counted number is an approximation, depending on overlapping particles, contamination in the system, etc.

6.2.3 Optional: Extraction

Particles can be extracted using vacuum. Extraction will aid dispersion of the particles and reduce contamination within the GRADIS. However, the sample is ultimately lost. Alternatively, without extraction, the particles can be collected at the base of the instrument (in a drawer) and re-used for other purposes.

6.2.4 Cleaning

Feeding using material >1 mm may produce (and measure) particle sizes <1 mm. This is caused by the mechanical effect of gravitational dispersion and the particle interaction: Particles (especially brittle material such as mica) may disintegrate and contaminate the system. The following are cleaning procedures at the department:

- Run a coarse “cleaning sample” (i.e. 2-4 mm) to use as a medium to extract smaller particles from the shaft. Remove contaminated particles from the extracted particles by performing a quick 1 mm sieving. This step may be repeated (repeat until the particle count after cleaning is as expected and stable).
- The GRADIS can be dismantled and cleaned with a vacuum cleaner, pressurized air etc. Due to the internal design cleaning can be difficult. The shaft must be mounted and aligned correctly before proceeding with measurements.
- Make sure to use a suitable outlet tip and check on a regular basis that particles can enter the measuring area freely.
- If the threshold value (see Signal Test window of the Windox software) is <70, additional cleaning of camera window etc. may be necessary. Without particles in the measuring area the threshold value should be >75.

Note that image segmentation is the process of partitioning a digital image into multiple segments. Pixels in a dynamic image are either *background* pixels or pixels belonging to the *foreground* (e.g. particles). The threshold value discriminates between pixels that are considered part of the foreground objects and pixels that are part of the background of the recorded images. To ensure that segmentation is carried out correctly, the threshold value must be monitored continuously.

6.2.5 Reliability of the method

It is crucial that particles are appropriately dispersed and fixed in the object plane of the instrument: The number of particles touching each other should be minimized. For this type of method, it is a prime objective that the measurements are carried out on isolated particles. Touching particles measured as one particle without proper separation will introduce errors. However,

it is often not possible to reliably detect touching particles by image evaluation alone, a great deal of user experience is needed.

To avoid the effects of velocity bias it is necessary that all particles traverse the measurement area at the same velocity. The selected frame rate has statistical relevance on the result. Both missed particles and multiple detection is possible, depending on the frame rate. Errors introduced by overlapping particles have a large effect on aspect ratio and length. As a consequence, the resulting PSD (and its characteristic values) may become coarser than it really is.

Image analysis on moving particles is somewhat different from static image analysis, there are some additional considerations:

- Any movement may create a motion blur effect which the image software must be able to process. To achieve the best result an optimum exposure time is crucial. This can be obtained by providing as much intensity as required for a sufficient contrast between background and particles, as long as the illumination is uniform over the total field of view.
- The movement must be aligned and positioned within the optical axis (depth of field). It is important that out-of-focus particles and other optical effects are minimized (otherwise these may introduce serious errors).
- The particles must be able to orient themselves freely with respect to the camera and relatively to each other.
- Particle images must be clearly distinguishable from the static background, aiming for threshold values >75.

Only objects within the image frame (field of view) should be accepted for measurement and counting.

If using reference materials for DIA they should be suitable for the technique and have known distributions and shape values. They must be easily dispersible and not exhibit electrostatic behavior.

6.3 Producing data

6.3.1 Evaluation

The system generates an rtf-file report (figure 65) that contains selected size and shape parameters based on a personalized template. The Department of Geoscience created a Python-executable to transform the text-information and import as data into a spreadsheet (Microsoft Excel compatible).

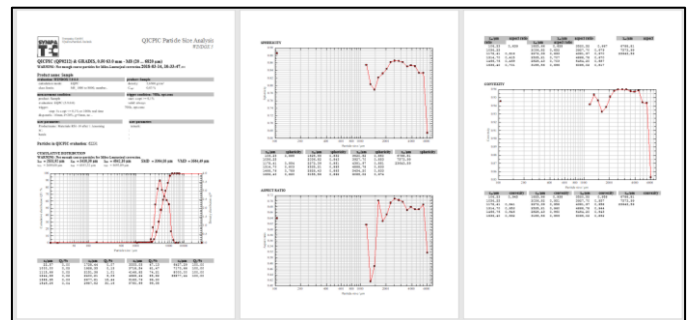


Figure 65: An example of a report containing size distribution and shape data (here cumulative distribution, sphericity, aspect ratio and convexity parameters).

Raw data can at all times be re-evaluated using different mathematical filters. In the Particle Gallery of the Windox software further image manipulations can be carried out. Here it is possible to setup filters for selected size and shape parameters (figure 66).

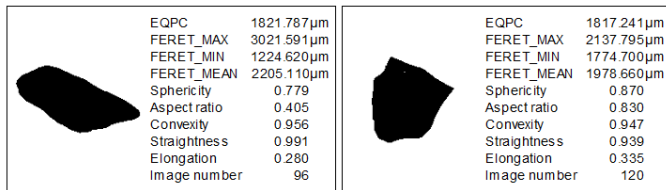


Figure 66: Example of applying filters to sort data provides size and shape information of single particles as well as a black and white image.

6.3.2 Sphericity

Sphericity S is the ratio of the perimeter P_{EQPC} of the equivalent circle with diameter X_{EQPC} to the perimeter P_{real} of the corresponding projection area A (figure 67). It follows that $0 < S < 1$, and a sphere has $S = 1$. The smaller the value, the more irregular is the shape of the particle.

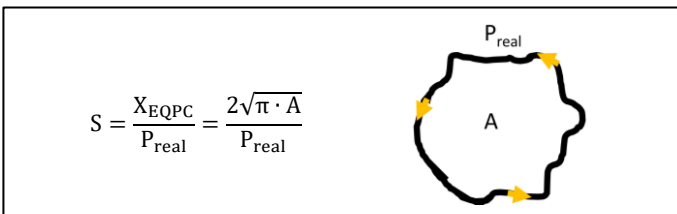


Figure 67: Definition of sphericity, P is perimeter and A is area. (After Sympatec)

6.3.3 Aspect ratio

Aspect ratio Y_A is defined as the ratio of the Minimum to the Maximum Feret Diameter (figure 68). It follows that $0 < Y_A < 1$. Note that particles with high aspect ratios have a tendency to show preferred orientations during DIA: Even with optimum dispersion and perfect measurement conditions their orientation may not be fully random.

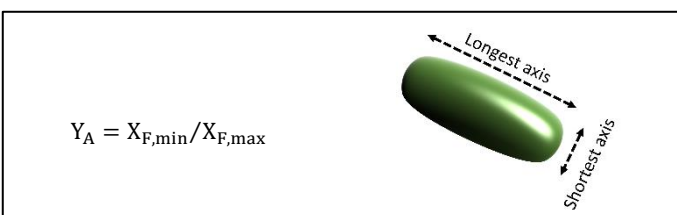


Figure 68: Definition of aspect ratio. (After Sympatec)

6.3.4 Convexity

The convexity ψ_c describes the compactness of a particle. Figure 69 shows a particle with projection area A (grey) leaving open a concave region of area B (red). The convexity is defined as the ratio of the projection area itself (A) and the area of the convex hull ($A+B$). The maximum theoretical convexity is 1, if there are no concave regions.

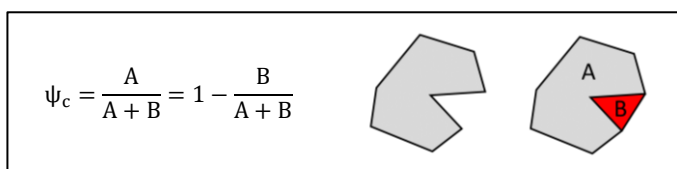


Figure 69: Definition of convexity. (After Sympatec)

Due to the detector design of a digital camera (square pixels), however, all particles seem to have small concave regions, corresponding to the tiny steps with every pixel in the perimeter line. Therefore, the maximum convexity calculated in reality is mostly limited to 0.99.

8 ADVANCED DATA MANIPULATION



8.1 Logarithmic computations

8.1.1 Aeolian sediments

From 1920 Ralph Alger Bagnold (figure 70) worked as a scientist and British army engineer in Libya and Egypt. While stationed in the deserts he investigated the physics of aeolian particles, moving through the atmosphere and deposited by wind: Aeolian sediments are dust, loess and sand. From this work the physics of sand transport has been developed. (Bagnold 1941)



Figure 70: Bagnold (left) was a pioneer, here he is inspecting the migrating coastal dune of Råbjerg Mile, Denmark, the largest moving dune in Northern Europe (photograph provided by Ole Barndorff-Nielsen, 2010).

The traditional interpretation of segmented curves in a probability diagram is often unjustified: 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) The parameters of the curves (and thus the interpretation of the distribution) can be better observed using the hyperbola instead of the parabola. (Christiansen, Blæsild and Dalsgaard 1984)

8.1.2 Parameters for log-hyperbolic distribution

The hyperbolic distribution requires several parameters for its specification, some describe the position and scale of the hyperbola and others describe the shape of the hyperbola. A combination of the parameters defines a hyperbolic log-size distribution, both visually and quantitatively: Roundness around the peak, sloping of the asymptotes and their interception (figure 71). (Bagnold and Barndorff-Nielsen 1980)

LOG-HYPERBOLIC DISTRIBUTIONS		
Symbol	Name	Parameter description
δ	Delta	Measure of roundness
μ	My	Peak diameter (μm)
ϕ	Phi	Slope, left linear asymptote (fine tail)
γ	Gamma	Slope, right linear asymptote (coarse tail)
κ	Kappa	Spread (an average of ϕ and γ)
ν	Ny	Mode of distribution (typical particle size)
τ^{-1}	Tau	Sorting (radius of circle inside curve)
χ	Chi	Approximate of skewness
ξ	Xi	Approximate of kurtosis (peakedness)

Figure 71: Description of the log-hyperbolic parameters. (Mikkelsen 2000-2004)

The domain of variation of the parameters χ and ξ is referred to as *the hyperbolic shape triangle*. The smaller the radius of the circle inside the triangle, the better the sample is sorted (figure 72). (Hartmann and Bowman 1993)

When estimating log-hyperbolic distributions the DOS software SAHARA has proven very useful. (Christiansen and Hartmann 1988) Note that the SAHARA software has certain limitations: Multimodal distributions cannot be processed and zero values are not accepted.

An alternative is GRADISTAT that only requires Microsoft Excel and allows for efficient transfer of data and statistics between other applications. It can be downloaded from the Internet: <http://www.kpal.co.uk/gradistat.html> (Blott and Pye 2001)

Licensed Windows software packages are also on the market (e.g. <https://www.ggu-software.com/en/geotechnical-software/>).

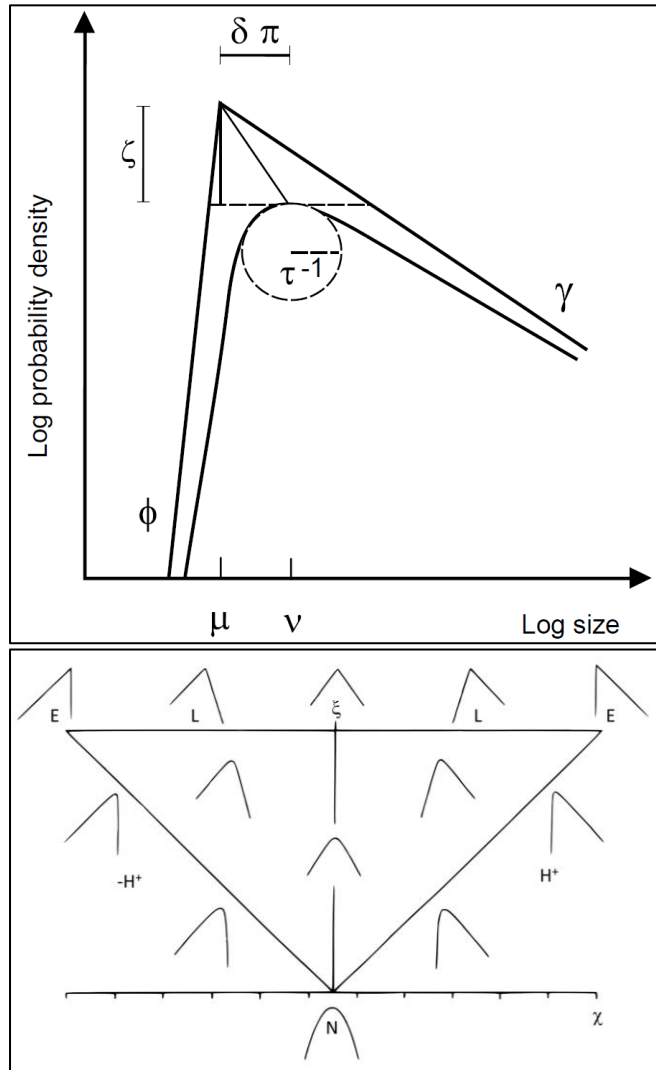


Figure 72: A geometric explanation of the parameters of the hyperbolic distribution (top). Below the log-hyperbolic shape triangle with logarithmic probability-functions corresponding to select values of (χ , ξ). (Dalsgaard 2006)

8.2 Miscellaneous equations

8.2.1 Calculating statistics and parameters

Often basic statistical parameters must be calculated to evaluate a size distribution. This is carried out by identifying typical size parameters such as D_{10} (the particle size where 10 % is below the size) or D_{90} (the particle size where 90 % is below the size), etc.

Note that the diameter of a particle size can be expressed in different ways. As an example, the particle diameter corresponding to the 10th percentile of the cumulative undersize distribution by volume: $D_{10} = D_{10,3} = \phi_{10} = D_v10 = D_{10} = x_{10}$. D_{10} is also called the *effective particle size*.

In the example $r(D_{10,3} r = 3)$ describes the quantity *type* and is a number ($r = 0$), length ($r = 1$), area ($r = 2$) or volume ($r = 3$).

Statistical parameters and graphic representations are usually given in ϕ units. Here are some of the most common values.

Sorting (figure 73) describes the spread of the distribution or the variation of particle size, also known as standard deviation (figure 74). Most soils are sorted, some however contain few variations, e.g. soil classified as “silt” can partially contain clay and fine sand. Till material usually contain considerable amounts of all fractions. Sorting values in the interval 1.0-2.5 is defined as well-sorted, 2.5-4.5 normally sorted and > 4.5 poorly sorted as first introduced by Trask in 1932. (Friedman 1962)

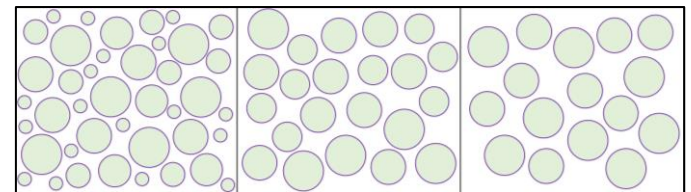


Figure 73: Sorting, ranges from poorly (left) to normally and well sorted (right).

$$\text{Sorting} = \sqrt{\frac{D_{25}}{D_{75}}}$$

Figure 74: A simple sorting expression for normal (Gaussian) distributions.

Other variations of sorting equations are also used (figure 75), the “inclusive graphic standard deviation” or σ_1 . Folk presented a verbal classification for this sorting: $\sigma_1 < 0.350$ very well sorted, 0.35-0.500 well sorted, 0.5-1.00 moderately sorted, 1.00-2.00 poorly sorted, 2.00-4.00 very poorly sorted and $\sigma_1 > 4.00$ extremely poorly sorted. (Folk and Ward 1957)

$$\sigma_1 = \frac{D_{84} - D_{16}}{4} + \frac{D_{95} - D_5}{6.6}$$

Figure 75: A more complex sorting expression, the “inclusive graphic standard deviation”.

The *mean* is a weighted average (center of gravity) and can be calculated by using the particle size at 25 % and at 75 % respectively (figure 76) for Gaussian distributions. Mean is the most widely compared parameter.

$$\text{Mean} = \frac{D_{25} + D_{75}}{2}$$

Figure 76: Mean, using two values.

This representation of mean is sensitive to the quantities of the extreme lower and upper ends of a distribution. Therefore, alternative methods to calculate the mean is also common such as M_2 (figure 77). This version may reflect bimodal and strongly skewed distribution curves more accurately. (Folk and Ward 1957)

$$M_2 = \frac{D_{16} + D_{50} + D_{84}}{3}$$

Figure 77: Mean, using three values.

The *median* corresponds to the 50 % percentile (50 % on a cumulative curve), where half of the particles are coarser and half of the particles are finer than this size (figure 78).

$$\text{Median} = D_{50}$$

Figure 78: Median.

The median can be a misleading value (any value based on one point only can be deceptive) and interpretation must be carried out with care.

The *mode* is the peak of the distribution or the most frequently-occurring particle size. For a symmetrical (normal) distribution the mode = median = mean (figure 79). Bimodal distributions have two modes (two distinct peaks) in the frequency distribution.

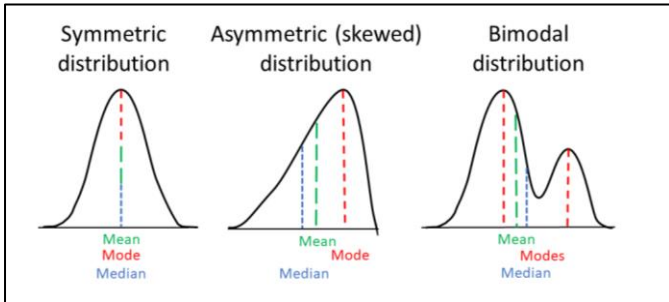


Figure 79: Examples of mean, median and mode on different distributions.

Skewness measures the degree to which the cumulative curve approaches symmetry, or the measure of deviation from symmetry (figure 80). Symmetrical curves have skewness 0. Different samples may have the same average particle size and similar sorting but be quite different in their degree of symmetry.

$$\text{Skewness} = \frac{D_{25} \times D_{75}}{\text{Median}^2}$$

Figure 80: A simple skewness expression.

Again, other variations of calculating skewness are available, some describe tailing (the extremes of the distribution) better such as the “inclusive graphic skewness” or sk_1 (figure 81). The verbal classification for sk_1 : +0.10 to -0.10 is nearly symmetrical, -0.10 to -0.30 is coarse-skewed and -0.30 to -1.00 is strongly coarse-skewed. (Folk and Ward 1957)

$$sk_1 = \frac{D_{16} + D_{84} - 2D_{50}}{2(D_{84} - D_{16})} + \frac{D_5 + D_{95} - 2D_{50}}{2(D_{95} - D_5)}$$

Figure 81: A complex skewness expression, the “inclusive graphic skewness”.

Kurtosis is the flatness or peakedness of the distribution (figure 82). Many curves designated as “normal” by the skewness measure turn out to be markedly non-normal when the kurtosis is computed.

$$\text{Kurtosis} = \frac{D_{75} - D_{25}}{2(D_{90} - D_{10})}$$

Figure 82: A simple kurtosis expression.

An alternative version of kurtosis is the “graphic kurtosis” or K_G (figure 83).

$$K_G = \frac{D_{95} - D_5}{2.44(D_{75} - D_{25})}$$

Figure 83: A complex kurtosis expression, the “graphic kurtosis”.

Gaussian curves have $K_G = 1.00$. On Gaussian curves sorting in the tails are equal to the sorting in the central portion. If a curve is better sorted in the central part than in the tails, the curve is said to be excessively peaked, or leptokurtic. If the curve is better sorted in the tails than in the central part, the curve is flat peaked or platykurtic (figure 84). The following verbal limits have been used for kurtosis: $K_G < 0.67$ very platykurtic, 0.67-0.90 platykurtic, 0.90-1.11 mesokurtic, 1.11-1.50 leptokurtic, 1.50-3.00 very leptokurtic and $K_G > 3.00$ extremely leptokurtic. In general, leptokurtic curves have $K_G > 1.00$ and platykurtic curves have $K_G < 1.00$. (Folk and Ward 1957)

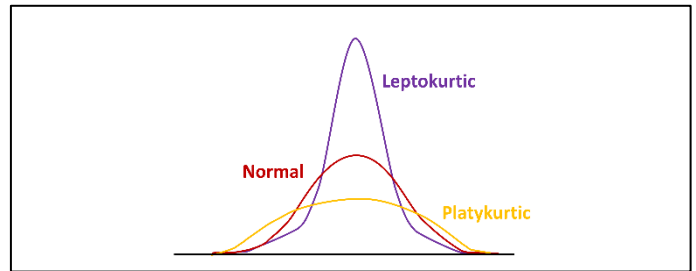


Figure 84: Examples of kurtosis.

Both skewness and kurtosis are vital clues to the bimodality of a distribution. Strictly unimodal distributions should have normal curves. Non-normal values of skewness and kurtosis indicate a mixing of two or more modal fractions. (Folk and Ward 1957)

Once all relevant parameters for a set of samples are calculated, it can be beneficial to select two parameters and plot them against each other in an (x, y) diagram to observe the trends.

8.2.2 Gradation

Another parameter sometimes used to describe material is the uniformity coefficient U or C_u (figure 85). A value of U < 2 classifies the particles as well-sorted, 2-3.5 as sorted, 3.5-7 as poorly sorted and U > 7 as unsorted. (Larsen, et al. 1995) Sometimes in international literature the equation is $U = D_{10}/D_{60}$ (which provides coefficients < 1).

$$U = \frac{D_{60}}{D_{10}}$$

Figure 85: The uniformity coefficient or U.

Related to the uniformity coefficient is the coefficient of curvature C_z or C_c (figure 86).

$$C_z = \frac{D_{30}^2}{D_{10} \times D_{60}}$$

Figure 86: Coefficient of curvature or C_z .

A soil sample is said to be well graded if it has all sizes of particles, where $1 < C_z < 3$. Poorly graded soil is material in which most of the particles are approximately of the same size, or uniformly graded. For uniform soil, $C_z = 1$.

Coarse-grained soils, mainly gravels or sands, are graded as either well graded or poorly graded. Poorly graded soils are further divided into uniformly-graded or gap-graded soils (figure 87). Soils that have some intermediate size or sizes not well represented, or at least one particle size missing, are called gap graded or skip graded. (Embuido, Azucena and Davis 1991)

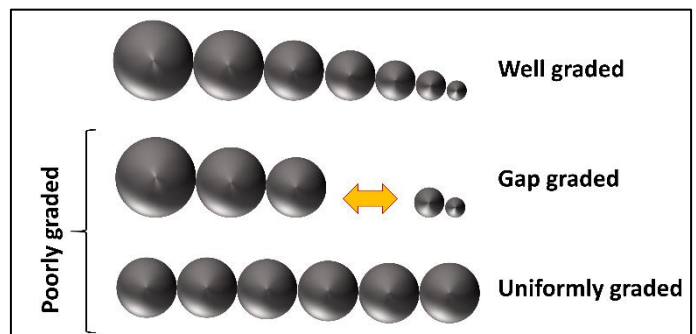


Figure 87: Different soil gradations.

For a *gravel* to be classified as well graded, the following criteria must be met: $U > 4$ and $1 < C_z < 3$. If both of these criteria are not met, the gravel is classified as poorly graded (GP). If both of these criteria are met, the gravel is classified as well graded (GW).

For a *sand* to be classified as well graded, the following criteria must be met: $U \geq 6$ & $1 < C_z < 3$. If both of these criteria are not met, the sand is classified as poorly graded (SP). If both of these criteria are met, the sand is classified as well graded (SW).

Soil gradation is an important aspect of geotechnical engineering and soil mechanics. It is an indicator of other engineering properties such as compressibility, shear strength, and hydraulic conductivity. Some examples:

- A well graded soil is able to be compacted more than a poorly graded soil.
- A poorly graded soil will have better drainage than a well graded soil because there are more void spaces.

8.2.3 Sortable Silt

Sediment enters the seas and oceans through various sources: Volcanic ash, aeolian dust, ice rafted debris, river runoff, etc. For marine sediments and in palae-oceanography the term sortable silt (SS) is of importance. Silt >0.010 mm displays size sorting in response to hydrodynamic processes and its properties may be used to infer oceanic bottom current velocity. (McCave, Manighetti and Robinson 1995)

The SS fraction is non-cohesive and therefore sortable by deep marine currents. If the site is far enough from land to not be influenced by e.g. river input or meltwater from glaciers, a larger SS value indicates strong currents at the ocean floor.

As flow speed increases, accumulation increases until a critical point is reached. After that point, the flow speed becomes too great and the flow begins to erode the sediment rather than depositing it.

The fraction of sortable silt, SS (%), is defined as the ratio of 0.010-0.063 mm grains in relation to all grains <0.063 mm. The second important parameter is the mean size of the sortable silt fraction, \bar{SS} (μm). This parameter is calculated as the sum of the products of the percentages and size of each class within 0.010-0.063 mm, divided by the total % of particles within 0.010-0.063 mm.

If SS is used along with tracers (such as isotope ratios), valuable information about the flow speed, the age of the body of water and chemical characteristics can be uncovered.

8.2.4 Interpolate particle size at specific percentile

Sometimes a percentile comparison diagram can be very useful: It is constructed from a set of percentiles (5, 10, 15 ... 95 %) by plotting particle size values, e.g. to compare two PSA methods. An equation is used to interpolate corresponding particle sizes at specific percentiles from each method (figure 88), taking into consideration that data is log-based.

$$\text{Size (Y)} = \left(\log(Y) - \log \left[y_1 \left(\frac{y_2}{y_1} \right)^{-x_1/(x_2-x_1)} \right] \right) \left(\log \left[\frac{y_2}{y_1} \right]^{1/(x_2-x_1)} \right)^{-1}$$

Figure 88: Two sets of particle sizes x (μm) and cumulated percentages y (%) are used for interpolation of particle size at a select percentile, Y . (Mikkelsen 2000-2004)

The equation looks complex but simply uses two coordinates (x_1, y_1) and (x_2, y_2) to interpolate a particle size at a select percentile, Y . This is repeated for relevant percentiles of each PSA method. It may be necessary to use some coordinates several times to interpolate different percentiles; it depends on how wide the distribution is.

As an example, PSA using the Andreasen pipette and LD has been performed on kaolinite. The material was carefully grinded and thus is expected to be fine. To test how data from the two methods compare, interpolation is carried out on each method, using their raw PSD data (figure 89 and 90). Differences in measured values are expected to be shape dependent.

Raw data of method 1 (Andreasen pipette), kaolinite	
Particle size [μm]	Cumulated percentage [%]
2	63.73
4	75.50
8	83.88
16	94.11
32	99.30

Figure 89: Cumulated raw data of kaolinite by Andreasen pipette.

Raw data of method 2 (LD), kaolinite	
Particle size [μm]	Cumulated percentage [%]
(0.5)	(0)
1.8	29.32
2.2	36.27
2.6	42.10
3.0	46.97
3.6	52.93
4.4	59.01
5.2	63.51
6.2	67.70
7.4	71.50
8.6	74.57
10	77.64
12	81.41
15	86.04
18	89.61
21	92.35
25	95.09
30	97.37
36	98.89
41	99.55
50	99.87
60	99.99
72	100
86	100
102	100
122	100
146	100
174	100
206	100
246	100
294	100
350	100

Figure 90: Cumulated raw data of kaolinite by LD (based on R4).

The two tables indeed show that the material has been measured as fine by both methods: According to method 2 (with the largest measured size) all particles seem to be < 0.060 mm.

Now the equation can be used to interpolate particle size values at percentiles, from 5, 10 etc. up to 95 %. In this example kaolinite is fine material and when evaluating the data, the percentiles to be used are in the range 65–95 % (the first cumulated percentage for method 1 is 63.73 %). E.g. if method 1 has (x, y) where x = particle size and y = cumulated percentage and the particle size at the 65 % percentile needs to be calculated, then select the closest relevant data points below and above 65 % from the raw data (figure 91) which is (x₁, y₁) = (2; 63.73) and (x₂, y₂) = (4; 75.50) respectively.

Interpolation, method 1 (Andreasen pipette) of kaolinite			
Per-centile [µm], Y	Particle size [µm], x	Cumulated percentage [%], y	Interpolated particle size [µm], X
65	2	63.73	2.23
	4	75.50	
70	2	63.73	3.11
	4	75.50	
75	2	63.73	3.92
	4	75.50	
80	4	75.50	6.20
	8	83.88	
85	8	83.88	8.92
	16	94.11	
90	8	83.88	12.90
	16	94.11	
95	16	94.11	18.82
	32	99.30	

Figure 91: Selected raw data for method 1 and calculating corresponding particle size X at each percentile Y using the interpolation equation.

The procedure is repeated for method 2 (figure 92).

Interpolation, method 2 (LD) of kaolinite			
Per-centile [µm], Y	Particle size [µm], x	Cumulated percentage [%], y	Interpolated particle size [µm], X
65	5.2	63.51	5.56
	6.2	67.70	
70	6.2	67.70	6.93
	7.4	71.50	
75	8.6	74.57	8.80
	10	77.64	
80	10	77.64	11.26
	12	81.41	
85	12	81.41	14.34
	15	86.04	
90	18	92.35	18.43
	21	92.35	
95	21	92.35	24.87
	25	95.09	

Figure 92: Selected raw data for method 2 and calculating corresponding particle size X at each percentile Y using the interpolation equation.

The calculated values of particle size from each interpolation table of the two methods are plotted in an arithmetic coordinate system, where i.e. the particle size for method 1 is plotted on the abscissa and the particle size for method 2 on the ordinate (figure 93). As an example, for the percentile at 65 % the coordinate becomes (2.23; 5.56). The rest is added likewise.

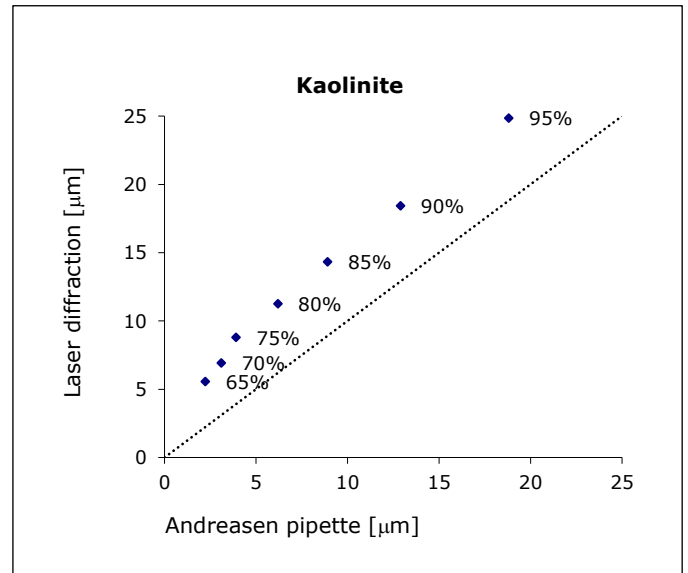


Figure 93: Creating a visual comparison between data obtained by two methods by using interpolation of particle sizes at specific percentiles. Here LD and sedimentation by Andreasen pipette are compared, the material measured is kaolinite.

Request a copy of a Microsoft Excel-template from the author to effortlessly interpolate log-based data as shown above.

9 CONCLUSIONS



9.1 Technical perspectives

9.1.1 Sampling

As is the case with all laboratory experiments and analysis, best-practice sampling and best-practice PSA is an important investment from day one.

All the time and effort put into one analysis is wasted, if sampling is poorly executed: Sampling needs care and attention. It is equally important to sub-sample carefully and measure representative material every time. It is essential to have a robust and tested protocol for sampling that describes sub-sampling and sample preparation, as well as having considered dispersion and measurement procedures that are suitable for the material.

Any systematic error may arise from poor sample preparation, departure from the theoretical assumptions for the material and the applied method, as well as improper operation of the equipment. Errors made in sample preparation are often the largest contributor to the total error, as well as the assumption that all particles are spherical.

Because of the differences in measuring particle properties, the obtained results should always be provided along with information about the sampling method, pretreatment and analytical procedure for others to be able to duplicate the result.

9.1.2 Quality of data

Reproducibility, standard deviations and accuracy are among the important factors to consider when qualifying a PSA procedure.

Precision and accuracy are two ways that scientists think about error. Accuracy is how close to the true value a given measurement is. Precision is how well a number of independent measurements agree with one another, in other terms: A measure of statistical variability (figure 94).

For PSD techniques it is recommended to ensure accuracy, by using traceable spherical certified reference materials (CRMs), e.g. particles with certified values. This ensures that instruments are correctly functioning. When any modification or major

maintenance is required, use CRMs to test accuracy of the instrument and quality of the PSD.

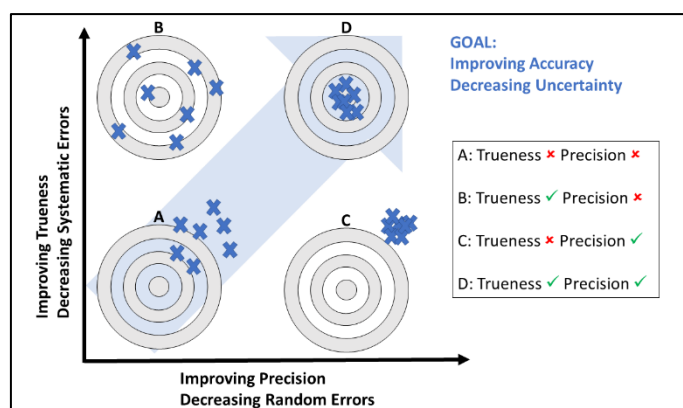


Figure 94: High accuracy and low uncertainty is desirable when evaluating data.

The straightforward strategy to increase precision in the laboratory and with PSA is by paying close attention to detail, using equipment properly and increasing sample volume.

Take each measurement multiple times, especially if performing experiments that require specific amounts (weight or volume). If weighing objects, remove them from the scale after the first reading and check the scale between the first and second readings. If using a beaker or pipette to measure volume, examine the approximate amount of liquid in the beaker while it sits still on a flat surface; check pipette settings between each use, etc.

9.1.3 Experimental factors

Again, when performing analytical experiments and analysis, many factors contribute to ensuring a fast and reproducible result of high-quality:

- The instrument/equipment and accessories
- Settings during each particular measurement
- The operator

In total: Available time, user experience, sample concentration (volume), dispersion, temperature, contamination, condition of the equipment, etc. Many factors contribute to the final result.

9.2 Other alternatives

9.2.1 NIR – for soil application

Another method not described in this paper but nevertheless interesting uses near-infrared (NIR) spectroscopy (or NIRS in short) to determine soil attributes, including chemical, physical and biological properties. NIRS for clay determination can be used directly on a bulk sample without pretreatment. NIRS can be used in the laboratory, in-situ and in remote sensing and is therefore a very flexible and non-destructive method for fine particles. (Cécillon, et al. 2009)

In short, this technique uses the near-infrared region of the electromagnetic spectrum to measure parameters that also deduce particle size. Chemical bindings (O-H, N-H and C-H) will absorb energy from light and reflect it by sending out radiation at a characteristic wavelength. Wavelengths in the mid-infrared region (MIR) is in the range 0.0025-0.025 mm and wavelengths in near-infrared region (NIR) is in the range 0.0007-0.0025 mm. (Knadel 2011)

Physical properties:

Particle size fractions, specific surface area, wet aggregate stability, physical surface crust.

Chemical properties:

Hygroscopic water, OM fractions (humic, fulvic acid), total N, mineral N, active N, P, pH, EC, CEC, K, Na, alkali earth metals (Ca, Mg), carbonate, sulfate, Al, Fe, Si, heavy metals (Mb, Zn, Cu, Co, Pb, Ni, Cd, Mo, Se, Hg, V, Cr, Ag).

Biological properties:

Methane production, enzyme activities, microbial respiration, microbial biomass, organic C, total N, C/N, inorganic C.

There are different NIRS instruments and they all have sample holders of different dimensions and offer different scanning methods. Some have large sample cups and for those it isn't necessary to do replications. By standard, the instrument scans in different areas of the material. For those with a smaller sample cup it is necessary to make additional replications. As an example, the FOSS sampling cup for model DS2500 (figure 95) has a capacity of approximately 50 g.



Figure 95: The Foss NIR table-top model as seen at Department of Agroecology, Aarhus University.

The FOSS instrument is not specifically designed for soil material. Normally 7 scans in different spots of the sample holder is performed and then a mean is calculated. For other commercially available NIRS sensors for soil, sampling cups can hold approximately 20 g of soil. Here is the option of doing measurements on

soil surfaces with a contact probe, not much material is needed: Approximately 5-10 g could be sufficient and replications are needed. For a sufficient number of sub scans and a relatively large sample volume the reproducibility is usually good. (Knadel 2020) Depending on the instrument resolution, 3-4 replicates have been shown to be the optimal number for MIR measurement. (Peng, et al. 2014)

Because of the complex nature of spectral data, the instrument must be calibrated on a series of natural samples representative of the population to be measured. Factors that affect the spectra are sample composition, particle size and moisture. NIRS methods must be validated continuously against reference methods to secure optimal performance and accuracy which is relatively resource demanding. When NIRS data is compared to data derived by traditional methods it shows a good compatibility. (Sørensen and Dalsgaard 2005)

A major advantage of using this method for soil analysis is that from a single spectrum many properties may be (accurately) determined, thus offering the possibility for considerable cost savings and increased efficiency over conventional laboratory analysis. Furthermore, the technique is still considered rapid, making it possible to analyze a large number of samples in a practical and timely manner. These properties make spectroscopic analyses very attractive for e.g. environmental monitoring and agriculture. (Viscarra Rossel, et al. 2006)

9.3 Final comments

9.3.1 Discussion

Different methods measure different parameters and data is thus not easily compatible, especially when dealing with complex variables such as unique shape and mineral composition.

When we improve our understanding of the physical context, we are often able to develop more advanced methods or models that fit the description of nature better. When evaluating traditional and new techniques, there seems to be no clear or universal relationship between the sieve or pipette methods and LD yet. One reason is the fact that the sieve and pipette methods measure mass percentages of particles whereas LD measures volume based on an optical diameter.

For all PSD techniques the basic idea of measuring each primary particle independently is vital. More investigations are needed on the boundary between clay and silt to clarify the mineralogy and particle shape dependencies. Since the clay limit is not fixed it would be beneficial to analyze fine silt in detail, since these particles exhibit some of the same properties as clay.

The use of LD, DIA and NIRS have many advantages: The methods are fast and offer a high reproducibility. A wide variety of parameters can be obtained within a short period of time, providing fast and detailed information. Care must be taken when matching historic data or correlate with other techniques or instruments.

Uncertainty errors arise when trying to compare data. Generally, most PSD techniques are most suitable for spherical particles of the same mono-mineral (which soil and sediment samples rarely are). Also, tiny variations in the techniques applied can affect the final PSD of any given sample; therefore, also differences in laboratory methods and pretreatment will affect the final data interpretation. (Cramp, et al. 1997)

9.3.2 Overview of methods

Different methods provide different options and opportunities to describe particle characteristics (figure 96). Choose the method that is best for the material and available resources as well as fulfills the immediate need for information.

	SIEVING	LD	SEDI	DIA
Theoretical validity [mm]	>0.075	0.001-6	<0.050	<6.820
Typical range [mm]	0.063-16	0.002-2	<0.063	1-4
Classes	10 (or more)	Infinite (in theory), 31 measured data-points (HELOS)	<5 (more could be added)	Infinite (in theory)
Medium	Dry	Wet, dry	Wet	Dry
Material [g]	150-200 (20 cm sieve)	5-150	5-8	500-5000
Destructivity	No	Yes and no	Yes	Yes and no
Pre-sieving	Yes	Yes	Yes	Yes
Reproducibility	Medium	High	Medium	High
User skills	Medium	Low	High	Low
Requirements	-	-	Temperature-control	-
Use	Laboratory, in situ	Laboratory	Laboratory	Laboratory
Acquirement [DKR]	<10000 + shaker	500000	<10000	500000

Figure 96: Comparing the different methods described in this paper.

All of the analyses described here are still relevant today. Although LD and DIA are considered more modern and technically advanced methods, and sieving and sedimentation analysis traditional, there is still a need and a market for the older methods too: They are simple, low-cost and relatively easy to learn and can fit in almost any setting.

9.4 Acknowledgements

Working with particle size distributions and quality of data processing and data interpretation have been huge motivation factors since 2003: They perfectly combine my interests within geology, laboratory and information technology.

Sincere thank you to my mentor and now associate professor emeritus Kristian Dalsgaard (Department of Geoscience, Aarhus University) for introducing me to particle characterization within geomorphology and soil physics: Kristian taught me traditional particle size methods and the physics behind them (Stoke's law, Brownian motion, van der Waal forces etc.). He trusted me with log-hyperbolic computations early on and let me experiment independently with the available PSA techniques.

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From Sympatec especially Kay Mootz, Stephan Sanetra and Sjoerd Sluimer have been very helpful in regards of instrument hardware solutions and configuration queries.

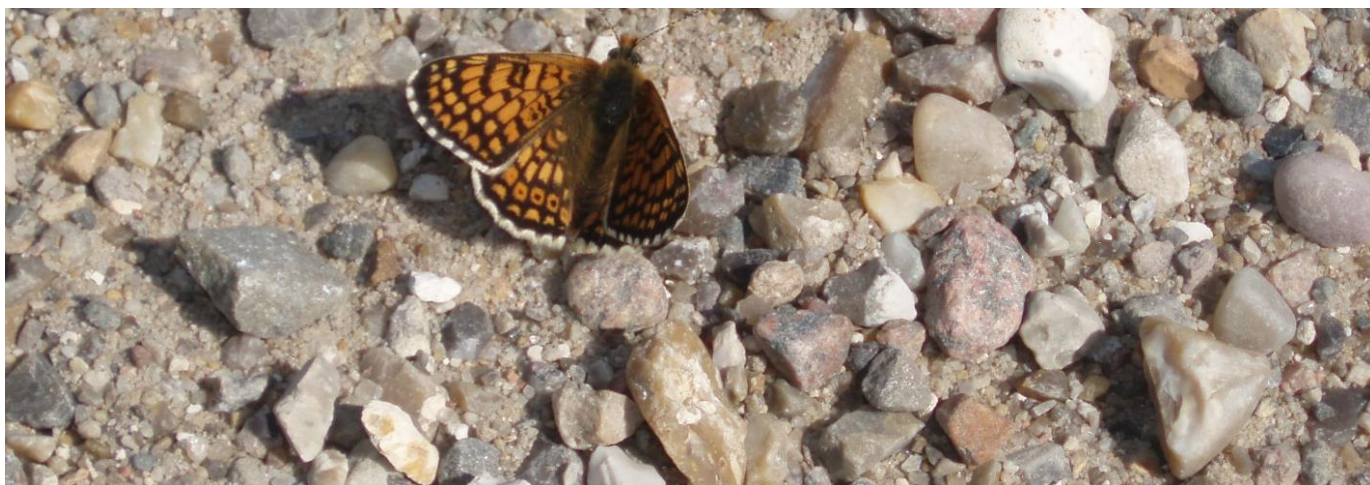
Working several years on the SESAM project I gained valuable hands-on experience working on sieving and sedimentation analyses on till, clay and sand samples: For the duration of the entire project over 4300 samples were processed. Simultaneously I managed the large data sets that was produced, including data obtained by X-ray diffraction and fine gravel (2-4 mm) analysis. For this I would like to thank associate professor emeritus Christian Kronborg and associate professor emeritus Ole Bjørnslev Nielsen (both Department of Geoscience, Aarhus University) who initially hired me into the position that ended up being permanent. Also, thank you to senior geologist Jette Sørensen (now VIA University College) for efficient teamwork and providing the basis for optimizing and managing the large data sets.

Recently Thomas Ljungberg created a python executable for the QICPIC to convert data stored as rich text into a suitable format. Many thanks for checking my chemical illustrations: Geochemist Gry Hoffmann Barfod (Department of Geoscience, Aarhus University) and professor emeritus Svend Erik Rasmussen (Department of Chemistry, Aarhus University). I reached out to my network for relevant SEM images, your fast response is much appreciated Margrethe Thorup Dalgaard. Geologist Anders Damsgaard quickly produced a nicely adaptable LaTeX equation.

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May 2020, Charlotte Rasmussen.

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APPENDIX

A: Wentworth grain size chart

Φ	PHI - mm CONVERSION φ - log ₂ (d in mm) 1 μm = 0.001 mm		SIZE TERMS (after Wentworth, 1922)	SIEVE SIZES		Intermediate diameters of natural grains equivalent to sieve size	Number of grains per mg		Settling Velocity (Quartz, 20°C)		Threshold Velocity for traction cm/sec		
	mm	Feet to nearest mm and Decimal inches		ASTM No. (U.S. Standard)	Tyler Mesh No.		Quartz spheres	Natural sand	Spheres (Gibbs, 1971) cm/sec	Crushed	(New in, 1946)	(modified from Hjulstrom, 1939)	
-8	256	10.1"	BOULDERS (≥ -8φ) COBBLES								200	1 m above bottom	
-7	128	5.04"											
-6	64.0	2.52"	PEBBLES	2 1/2"	2"								
-5	53.9	1.26"		very coarse	1 1/2"	1 1/2"							
-4	45.3			coarse	1 1/4"	1.05"							
-3	33.1	0.63"		medium	3/4"	.742"							
-2	32.0			5/8"	1/2"	.525"							
-1	26.9			3/8"	5/16"	.265"							
0	22.6	0.32"		fine	4	4							
-1	17.0				5	5							
-2	16.0	0.16"		very fine	6	6							
-3	13.4				7	7							
-4	11.3	0.08"	Granules	8	8								
-5	9.52			10	10								
-6	8.00	0.08"	very coarse	12	10								
-7	6.73			14	12								
-8	5.66	0.08"	coarse	16	14								
-9	4.76			18	16								
-10	4.00	0.08"	medium	20	20	1.2	.72	.6	10	7	40	40	
-11	3.36			25	24	.86	2.0	1.5	10	7	30	30	
-12	2.83	1/2	fine	30	28				8	6			
-13	2.38			35	32	.59	5.6	4.5	8	6			
-14	2.00	1/4	very fine	40	35				7	5			
-15	1.63			45	42	.42	15	13	7	5			
-16	1.41	1/8	coarse	50	48				6	4			
-17	1.19			60	60	.30	43	35	6	4			
-18	1.00	1/16	medium	70	65				5	4			
-19	.840			80	80	.215	120	91	5	4			
-20	.707	1/32	fine	100	100				4	3			
-21	.545			120	115	.155	350	240	4	3			
-22	.500	1/64	very fine	140	150				3	3			
-23	.420			170	170	.115	1000	580	3	3			
-24	.354	1/128	coarse	200	200				2	2			
-25	.297			230	250	.080	2900	1700	2	2			
-26	.250	1/256	medium	270	270				1	1.0			
-27	.210			325	325				1	1.0			
-28	.177	1/512	fine	400	400				0.5	0.5			
-29	.149								0.5	0.5			
-30	.125	1/1024	very fine						0.1	0.085			
-31	.105								0.1	0.085			
-32	.088								0.023				
-33	.074								0.01				
-34	.062								0.0057				
-35	.053								0.0014				
-36	.044								0.001				
-37	.037								0.00036				
-38	.031								0.0001				
-39	.016												
-40	.008												
-41	.004												
-42	.002												
-43	.001												

(Wentworth 1922)

Note: The relation between the beginning of traction transport and the velocity depends on the height above the bottom that the velocity is measured, and on other factors.

Note: Some sieve openings differ slightly from phi mm scale

Note: Sieve openings differ by as much as 2% from phi mm scale

Note: Applies to subangular to subrounded quartz sand (in mm)

Note: Applies to subangular to subrounded quartz sand

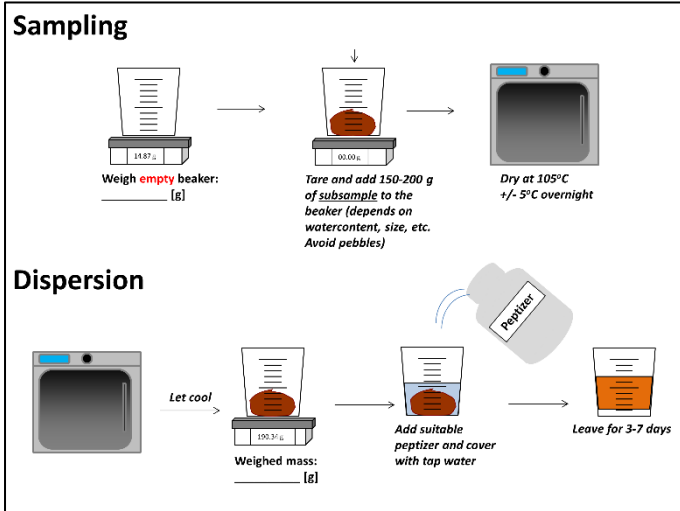
Stokes Law (R = 6πrηv)

Minimum (Inman, 1949)

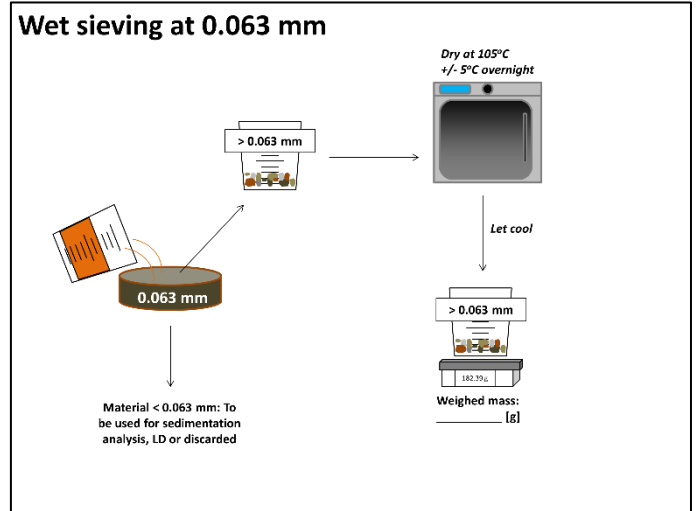
B: Flow chart sieving

The entire procedure for sieving analysis is described here, without pretreatment (refer to chapter 2).

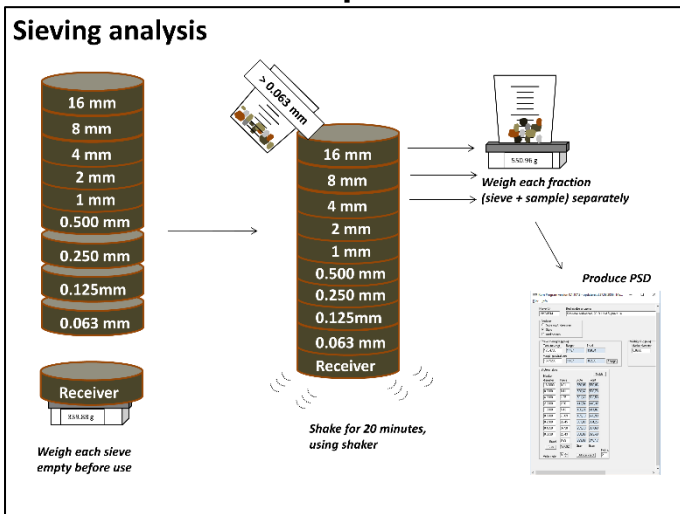
Step 1



Step 2



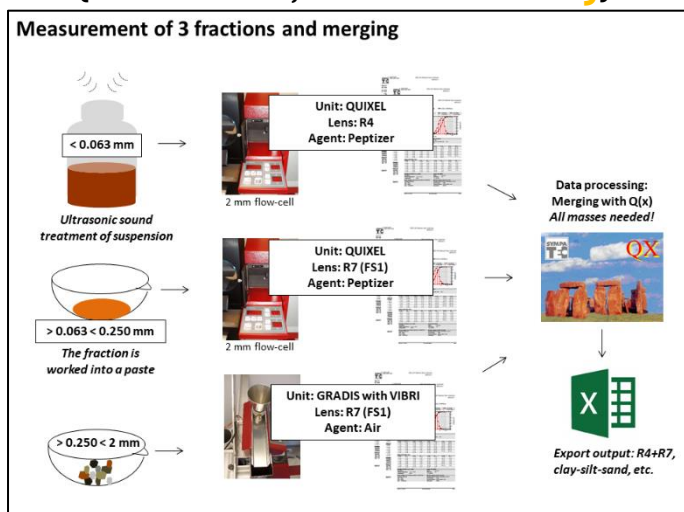
Step 3



C: Flow charts LD

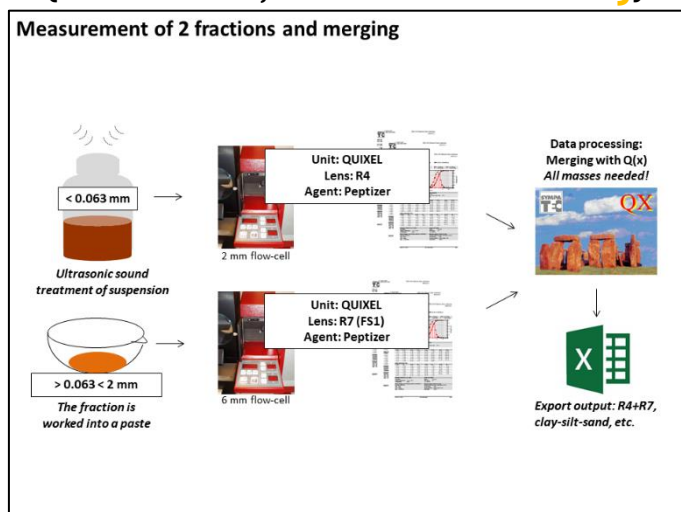
Refer to pretreatment procedures in chapter 2 and detailed description of LD measurements in chapter 4. Note that "Standard procedure < 2 mm" is described thoroughly in chapter 4 and the four other versions shown here are variations of this procedure.

1: Standard procedure < 2 mm (3 fractions, start mass 50 g)



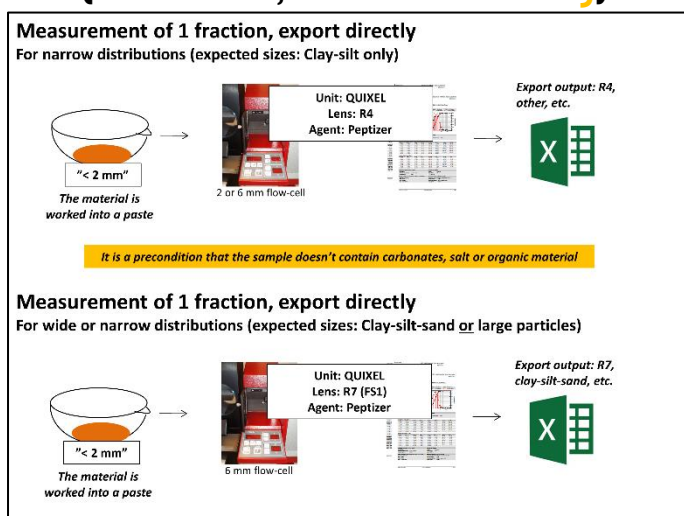
Standard procedure for soil and sediment samples at the Department of Geoscience. Note that Sympatec's standard method is limited to two fractions, splitting at 0.063 mm and working < 0.063 mm into a paste. Then using GRADIS for > 0.063 mm.

2: Material < 2 mm (2 fractions, start mass 10-20 g)



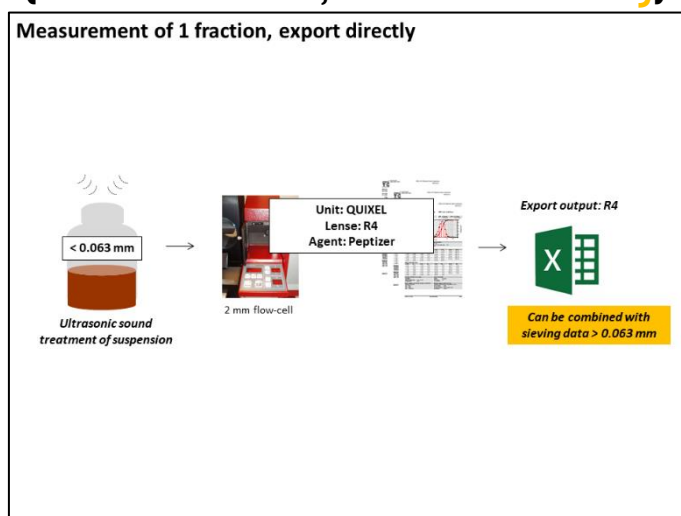
"Poor man's" version of the standard procedure for soil and sediment samples: You want to separate the material into coarse and fine particles, but can only afford to do two fractions. Useful when only small volumes of material are available or time is limited.

3+4: Material "< 2 mm" (1 fraction, start mass < 5 g)



Two variations when there is only little material available. Make sure that the material is < 2 mm, this can often be done visually or by doing a quick 2 mm sieving at the instrument. Useful for marine core material.

5: Material < 0.063 mm only (1 of 2 fractions, start mass 150 g)

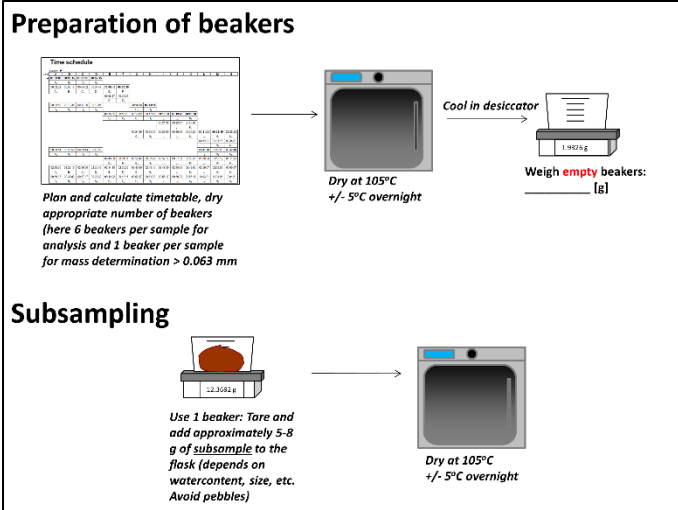


This version assumes that you have other means of determining particle size > 0.063 mm but want to ensure a quick PSA < 0.063 mm by LD. Used for lab-courses of till material where > 0.063 mm is sieved.

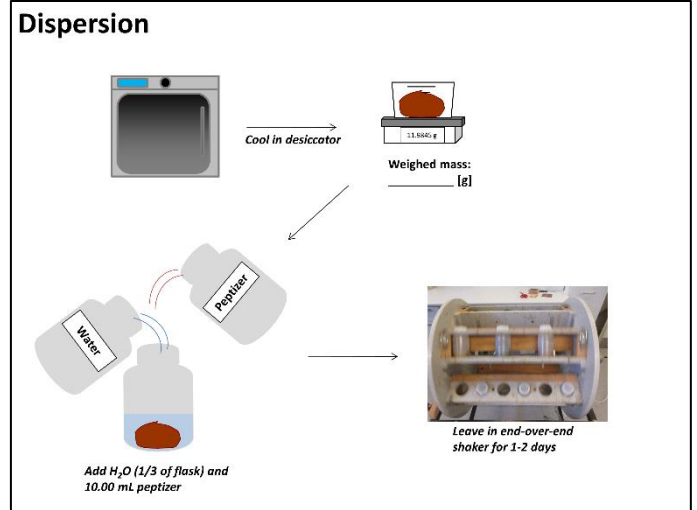
D: Flow chart sedimentation

The entire procedure for sedimentation analysis is described here, normally there is no further pretreatment.

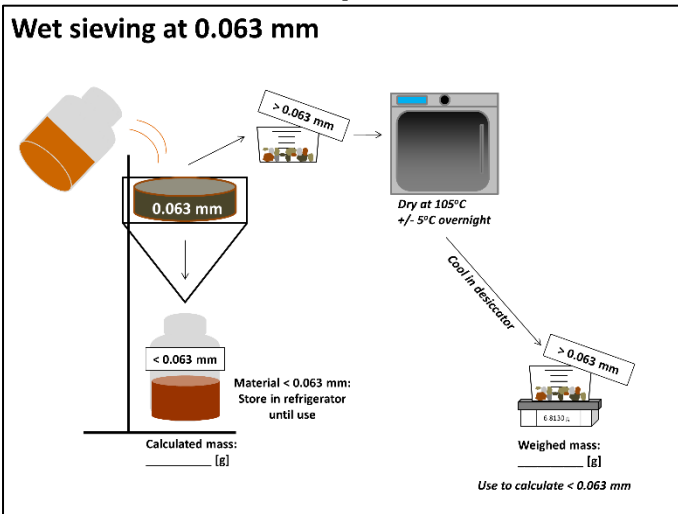
Step 1



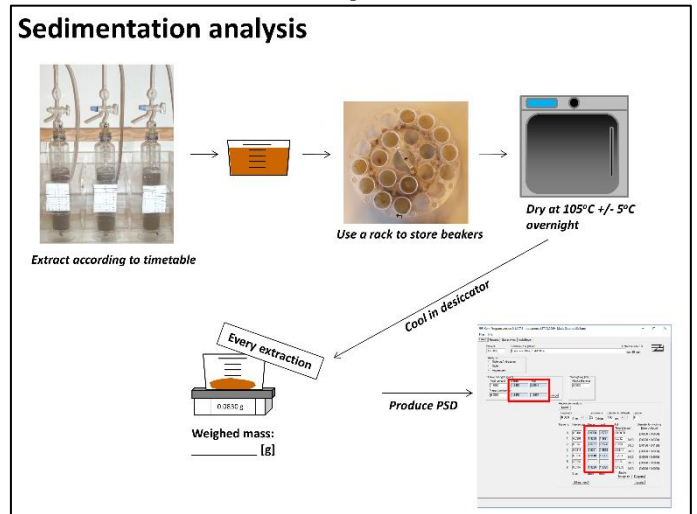
Step 2



Step 3



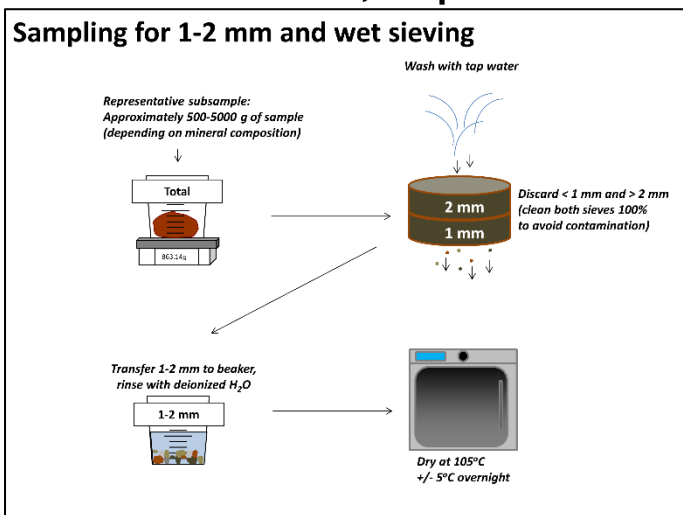
Step 4



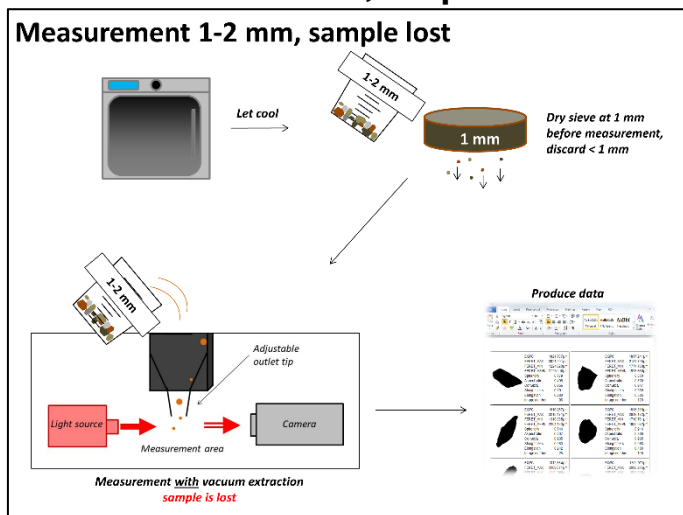
E: Flow charts DIA

The entire procedure for DIA is described here in two versions.

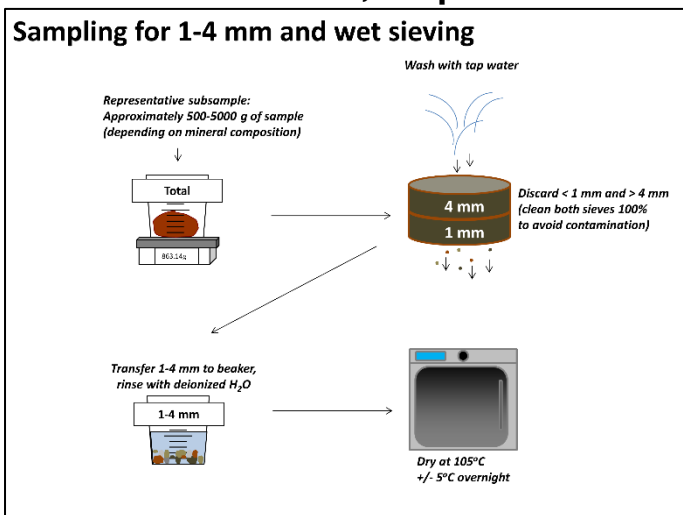
Version 1, step 1



Version 1, step 2



Version 2, step 1



Version 2, step 2

