

Comparison of grain size distribution methods applied to Halden silt

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Acknowledgments

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Introduction

Particle-size distributions (PSDs) are fundamental physical properties of soil. Typically, they are presented as the percentage of the total dry weight of soil occupied by a given size fraction. This property is commonly used for soil classification and estimation of some hydraulic properties.

Over recent decades, various methods for grain-size analysis have been developed. These methods are for example hydrometer analysis, falling drop analysis, electro-resistance particle counting, time of transition, laser diffraction (LD) and optical determination of the PSD using image analysis. Some of these methods generally have the advantage of covering a wide range of grain sizes and rapidly analyzing small samples.

Most of the methods for grain-size analysis are based on the assumption that the sedimentation process follows Stokes' law which is based on measuring sedimentation speed-equivalent diameters during the sedimentation process. Some other methods, based on image analysis, are based on a plan-view of particle diameters. This can by definition give different results for PSDs.

For silty soils, the definition of the clay content is crucial for soil classification. The experience with hydrometer falling drop analysis have shown PSDs for silty soils with more than 10% difference in the clay content. In Norway clay is often considered to be a sediment with at least 30% clay-sized grains, i.e. grains smaller than 2 microns. The remainder of the sediment is often dominated by silt, which has grains between 2 and 63 micrometers. There is an urgent need in identifying the aspects that control the consistency and differences in interpretation of grain-size analysis for silty soils.

The purpose of this project is to perform grain-size analyses on the same material by three different methods: hydrometer, falling drop and Coulter, in order to quantify the mechanisms that give similarities and differences for PSDs in silts. Samples from the Halden silt site, which is part of the Norwegian GeoTest Site project, have been used in the study.

CHAPTER 1

1.1 Background

Particle size distribution measurement has a long history. Lea and Nurse (1947) have published results on a symposium specifically organized for exchanging knowledge on particle-size analyses in 1947. The hydrometer (also called areometer) method was one of the first methods to be introduced (Bouyoucos, 1962). The original patent most likely was published in the USA, filed in 1959 and printed in 1964 (Rich, 1964). In 1965, the methodology of particle size analyzes was also published in the USA (Day, 1965). There are several detailed descriptions of this methodology available in literature explaining soil particle size measurements as part of normal soil analysis methods (Tan, 1996) or describing particle size measurements in general (Rich, 1964). The hydrometer method (also known as aerometer method) is based on settlement times and periodic density measurement. An hydrometer is an air filled glass tube that is able to measure density based on buoyancy in the suspension. The depth of immersion and the measured suspension's density in each trial allows for the opportunity to calculate the actual particle's diameter and the adherent weight in percent.

The classical pipette and hydrometer (aerometer) methods have been dominant in laboratory practice for several decades. Even though, hydrometer is a simple and fast method, the pipette is more accurate (Naguib and Bedaiwy, 2012). Due to the fact that both methods are time consuming, loaded with some errors and need a large samples (at least 10 g) of soil; the last 10-15 years has brought new techniques (e.g. laser diffractometry) that have begun to succeed the previous ones (Andrenelli et al., 2013; Beuselinck et al., 1998).

The two classes of methods of determining the particle size distribution (PSD) of a given sample consist of

- Classical (sieving and pipette methods)
- Instrumental (optical determination of particles, electrical sensing zone or electro-resistance particle counting (Coulter Counter), X-ray sedimentation (Sedigraph) and laser diffraction)

1.2 Methods

1.2.1 Laser diffraction Coulter method

The Coulter method is based on laser diffraction. The laser must not be confused with Coulter Counter, which is based on a measurement principle which is considered by some to be unsuitable for grain distribution analyzes of clay soils (Norges Geotekniske Institutt, 1986).

The laser diffraction method (LDM) is fairly new. The principle of LDM is that particles of a given size diffract light through a given angle. The angle of diffraction is inversely proportional to particle size, and the intensity of the diffracted beam at any angle is a measure of the number of particles with a specific cross-sectional area in the optical path. A parallel beam of monochromatic light passes through a suspension contained in a sample cell, and the diffracted light is focused onto detectors. For calculating particle sizes from light intensity sensed by detectors, two diffraction theories are commonly used: the Fraunhofer diffraction and the Mie theory. Both theories assume that the particles have a spherical shape; in other words, the particle dimension is the optical spherical diameter, i.e. the diameter of the sphere having a cross-section area equivalent to the measured one by laser diffraction.

Test execution:

Coulter LS200 is suitable according to the manufacturer for the determination of grain sizes in the range of 0.4 μm to 500 μm (can be used up to 2000 μm). The sample material is dissolved in water, added dispersant and placed in the apparatus. A laser beam is sent through an upstream flow of flow and will break at certain angles depending on the size of the particles. The angle of the broken rays is detected by up to 130 detectors ("channels") that correspond to given particle sizes. The intensity detected by the corresponding detectors is related to the number of particles of a given size. Grain breakdown is determined by volume. Assuming that the minerals in the different Fractions have the same density, cumulative volume percentages will be identical to cumulative mass percentages. Flake-shaped particles (clay minerals), where the particle size varies after the orientation of the particles relative to the laser beam, will be recorded with an average value assuming the diffraction is caused by spherical grain shape ("optical diameter"). The result will therefore be the underrepresentation of the finest material in relation to the methods based on the sedimentation principle.

1.2.2 Stokes law – Hydrometer

The hydrometer method is based on Stoke's Law that establishes the velocity at which particles settle in suspension assuming that:

- soil particles are rigid, spherical and smooth
- soil particles have similar densities
- particle-to-particle interference and boundary effects from the walls of the sedimentation column are negligible
- particle sizes are small enough to ensure that the induced fluid flow is well within the laminar flow regime.

A particle size calculated by Stokes' law is the quartz equivalent spherical sedimentation diameter. Deviations from Stokes' law are expected when particles are irregular in shape, as most silt particles, or are platy or tubular in shape as are most clay particles. The particle-shape effect is due to the circumstance that 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. As a consequence, this position increases the expected particle drag resistance and reduces the settling velocity. In other words the particle-shape effect results in a so-called "overestimation" of the fine size fraction which depends on at which size the platy particles appear.

Test execution:

- Place the hydrometer in the reference solution ensuring it floats freely. Take the hydrometer reading ($R'0$) at the upper rim of the meniscus to the nearest 0.0005 g/ml. Remove the hydrometer and rinse it well with distilled water.
- Agitate the water suspension vigorously until full suspension is obtained.
- Start the timer at the instant the agitation is complete.
- Promptly place the cylinder in its test position without any further delay.
- Take hydrometer's reading (Rh') at the upper rim of the meniscus after short periods of time. Take at least three measurements during the first 5min.
- After these initial readings, remove the hydrometer slowly and rinse it well with distilled water.
- At least three more readings are required. Readings at 4 min, 8 min, 30 min, 1h, 2h, 6h and 24 hours are often found to be suitable.
- The test may be stopped when the proportion finer than 0.002 mm has been determined.
- Record the temperature of the suspension once in the first 15 min and then after every hydrometer reading.

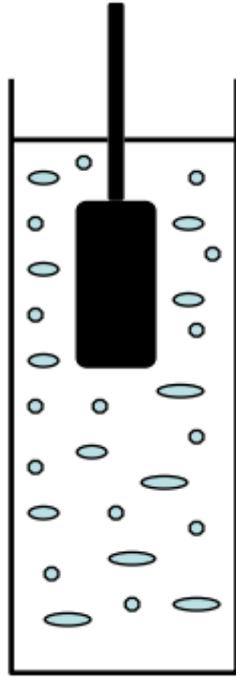


Figure 1: Schematic diagram of hydrometer test

1.2.3 Stokes' law - Falling drop

The falling drop method was worked out by the Norwegian Geotechnical Institute in 1964. The method is primarily a sedimentation method based upon Stokes' law. Utilizing this law for grain-size analyses requires, as is well-known, determination of the density of the particle suspension at certain depths of the sedimentation vessel after calculated sedimentation times.

Test execution

By using a calibrated micro-syringe or a micro-pipette a drop of the particle suspension is sampled at the desired depth of the sedimentation vessel, according to the corresponding calculated time for the grain-size in question. The drop is then ejected into a column of an organic liquid and the falling time over a certain distance is measured with a stopwatch. The concentration of suspended particles can then be read from a calibration chart, as shown in Fig.2. As organic liquid anisole ($C_6H_5OCH_3$) was chosen. At room temperature this has a density slightly less than water at the same temperature. To construct the calibration chart (Fig.2) drops of different sodium- chloride solutions with known densities were ejected into the column and the time for a 40 cm falling distance measured. The densities are drawn on a linear scale and the times on a hyperbolic scale. If the fall of the drops had followed Stokes' law, the curves then should have been straight lines, but as is seen, there are deviations.

The conversion from densities to particle concentration is achieved by using the formula:

$$\gamma_s = c \left(1 - \frac{\gamma_L}{\gamma_P}\right) + \gamma_L$$

where γ_s = density of sodium-chloride solution in g/cm^3

c = particle concentration in g/cm^3 for a particle suspension with density

γ_s ; γ_L = density of suspension liquid (i.e., water or water + dispersant) in g/cm^3

γ_P = density of particles in g/cm^3 .

From the particle concentrations c the amount less than a stated diameter d could be found by $\%< d = c_t * 100/c_0$ where c_0 and c_t are the concentrations of particles in the sedimentation vessel at the chosen depth respectively at the start of sedimentation and after a time ct , t and d being given by Stokes' law.

As further seen from Fig.2, the temperature control is very important. In the concentration range 40-50 g/l a temperature change of 0.1°C would give an apparent change in concentration of 0.25 g/l (about 0.6%) and correspondingly in the concentration range 2-7 g/l an apparent change of 0.12 g/l. A temperature control better than 0.1°C is, however, easily obtainable with proper thermostatic equipment.

The size of the drops used have been 40 mm^3 and the accuracy in sampling and ejecting these drops by means of a micro-syringe or a micro-pipette is about 1 mm^3 . Fig.3. shows how measured falling times vary with drop size for different densities of the drop.

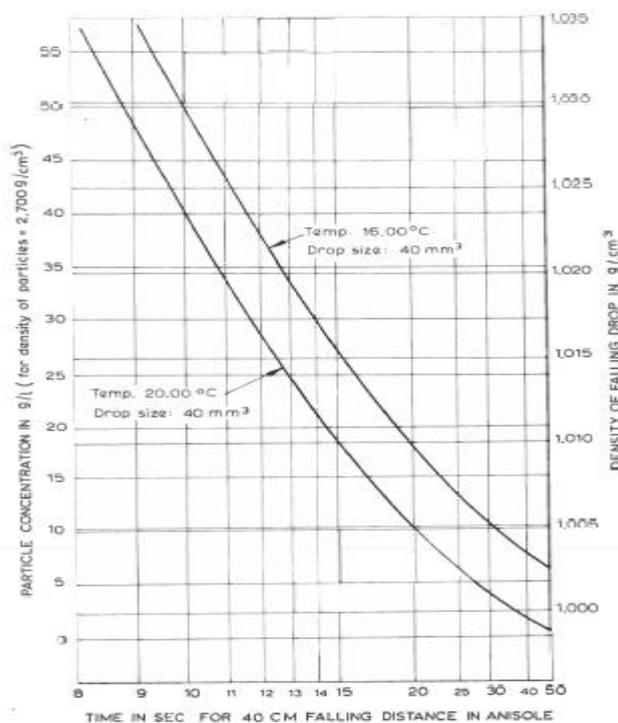


Figure 2: Calibration chart drawn on hyperbolic paper.

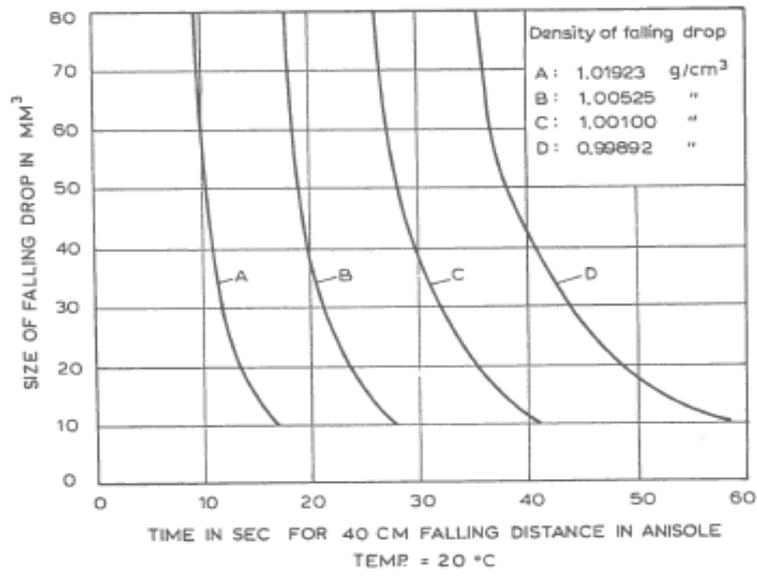


Figure 3: Influence of drop size on falling times for drops of different densities.

1.3 Comparison between methods for grain size analysis

Table 1 compares three methods applied in the present project to determined PSDs. The items evaluated in the comparison are further explained below:

COST: The price to obtain the required equipment.

PRINCIPLE: The basic theory behind the test

BEST FOR: More suitable material where the method can be applied.

FRACTION SIZE: The appropriate portion of a sample composed of particles between two given size limits that is suitable for each test.

SAMPLE WEIGHT: The amount of sample used in the test procedure.

RUNNING TIME FOR SAMPLES: The time it takes to finish the test procedure and acquire the results.

PROCESSING DATA: The way of handling the data during the test.

DRAWBACK: An important disadvantage of the test.

Table 1: Comparison between the three methods to PSD determination.

	Hydrometer	Falling drop	Coulter
Cost	Low cost :60NOK	Low cost: 155 NOK	Expensive \$12,500.00
Principle	Stoke's law (sedimentation)	Stoke's law (sedimentation)	Laser diffraction
Best for	Fine particles	Silt and clay	Sand materials
Fraction sizes	Fine particles <63 μm	<75 μm	0.4 μm to 500 μm
Sample weight	30-40 gr	1-5 gr	1 gr
Running time for samples	Intermittent readings over long period (5,30h) Results after 22hours.	10 minutes	10 minutes
Drawback	overestimation of the fine size fraction due to the particle-shape effect		underestimates the ratio of clay fraction (<2 μm) because of the lamellar shape of some clay minerals and because of the refractive index effect

1.4 Additional methods

Water content determination

The water content was obtained by the following method:

1. Clean a container with lid dry it and weigh it (W₁). Make sure you do this after you have tared the balance.
2. Take a specimen of the sample in the container and weigh with lid (W₂).
3. Keep the container in the oven with lid removed. Dry the specimen to constant weight maintaining the temperature between 1050 C to 1100 C for a period varying with the type of soil but usually 16 to 24 hours.
4. Record the final constant weight (W₃) of the container with dried soil sample.

The water content of the sample is $W (\%) = [(W_2 - W_3) / (W_3 - W_1)] \times 100$.

Specific gravity determination by pycnometer.

- (1) Determine and record the weight of the empty clean and dry pycnometer, WP.
- (2) Place 25 g of a dry soil sample (passed through the sieve No. 10) in the pycnometer. Determine and record the weight of the pycnometer containing the dry soil, WPS.
- (3) Add distilled water to fill about half to three-fourth of the pycnometer. Soak the sample for 10 minutes.
- (4) Apply a partial vacuum to the contents for 10 minutes longer, to remove the entrapped air.
- (5) Stop the vacuum and carefully remove the vacuum line from pycnometer.
- (6) Fill the pycnometer with distilled (water to the mark), clean the exterior surface of the pycnometer with a clean, dry cloth. Determine the weight of the pycnometer and contents, WB.
- (7) Empty the pycnometer and clean it. Then fill it with distilled water only (to the mark). Clean the exterior surface of the pycnometer with a clean, dry cloth. Determine the weight of the pycnometer and distilled water, WA.
- (8) Empty the pycnometer and clean it.

Specific Gravity, $G_s = W_0 / (W_0 + (WA - WB))$

Where: W_0 = weight of sample of oven-dry soil, $g = WPS - WP$

WA = weight of pycnometer filled with water

WB = weight of pycnometer filled with water and soil.

CHAPTER 2

2.1 SAMPLE ORIGIN AREA

The soil used in this project is originating from Halden, a thick deposit of fjord marine silt in south-eastern Norway. Halden is located in south-eastern Norway, approximately 120 km south of Oslo. Deposits at the area consists of marine and fjord marine sediments that emerged from the sea following a fall in relative sea level in the Oslofjord region during the last c. 11 000 years. During the post-glacial period, the depositional environment mainly led to hemipelagic deposition in a fjord marine environment. Due to the steady isostatic uplift in the Holocene and the fact that the sediments deposited continuously during a simple period of submergence , the soils in the area are expected to be essentially normally consolidated except for some surface weathering.



Figure 4 : Map of Halden (Google Maps)

2.2 Sample preparation

The 54mm diameter cylinder samples we had to test were from the borehole HALB03 in Halden and each of them had a length of 80 cm. We had five cylinders from different depths and we choose to test three of them. Our cylinders were :

- Cylinder 1 : Depth 3 to 3,8 meters.
- Cylinder 4: Depth 6 to 6,8 meters.
- Cylinder 11: Depth 13 to 13,8 meters.

Samples were stored in the laboratory vertically under controlled temperature conditions (ca. +50C) and high moisture content. The first step was to extrude the samples. Extrusion of undisturbed piston samples is carried out by specially designed hydraulic sample extruders. During this procedure the sample should be handled with extreme caution to avoid any disturbance. Bending or tension of the sample should be avoided.

Visual observation of cylinders:

Cylinder 1: Grey color with some organic material (roots and some fraction of wood).

Water content w : 15.2%

Density of grain soil $\rho_s = 2.6 \text{ g/cm}^3$

Cylinder 4 : Grey color.

Water content $w = 29\%$

Density of grain soil $\rho_s = 2.64 \text{ g/cm}^3$

Cylinder 11 : Grey color.

Water content $w = 25.2\%$

Density of grain soil $\rho_s = 2.66 \text{ g/cm}^3$



Figure 5 : Extraction of cylinder 1 (personal archive)

2.3 Mixing of the sample

The sample from each cylinder was used for three types of test (Coulter, Hydrometer and falling drop). So the soil had to be mixed in order to be as homogenous as possible before been tested and to dismiss sources of variation in the results. The procedure followed for all the cylinders was the following:

- Each cylinder sample was put in a steel mixing bowl and mixed manually for 2 minutes.
- Then, a laboratory mixer was used for 2 minutes at low speed.
- If the sample didn't look homogenous after this time, the sample was then mixed for other 2 minutes with a spatula as gently as possible.
- Finally, the samples were separated in equal proportions to perform each test: 3 hydrometer analysis, 1 falling drop and 1 Coulter analysis pr. Depth (i.e. pr. cylinder).

2.4 Determination of PSD

The hydrometer analysis was performed by me at NTNU and in accordance with the ISO 17892-4 as described at chapter 1.2.2. The three hydrometer tests per depth are shown in the appendix.

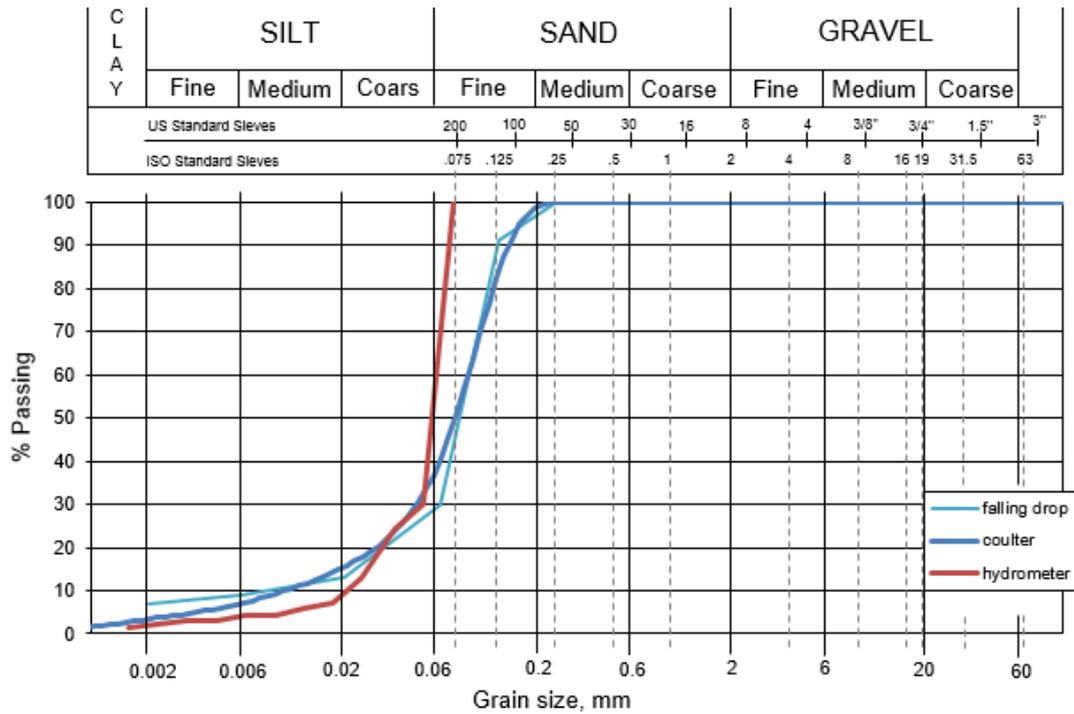
The Falling drop analysis was performed at NGI and the results are presented at the appendix.

The laser diffraction method was performed at NGU and the results are presented at the appendix.

CHAPTER 3

3.1 RESULTS

Cylinder 1 : 3-3,8 m

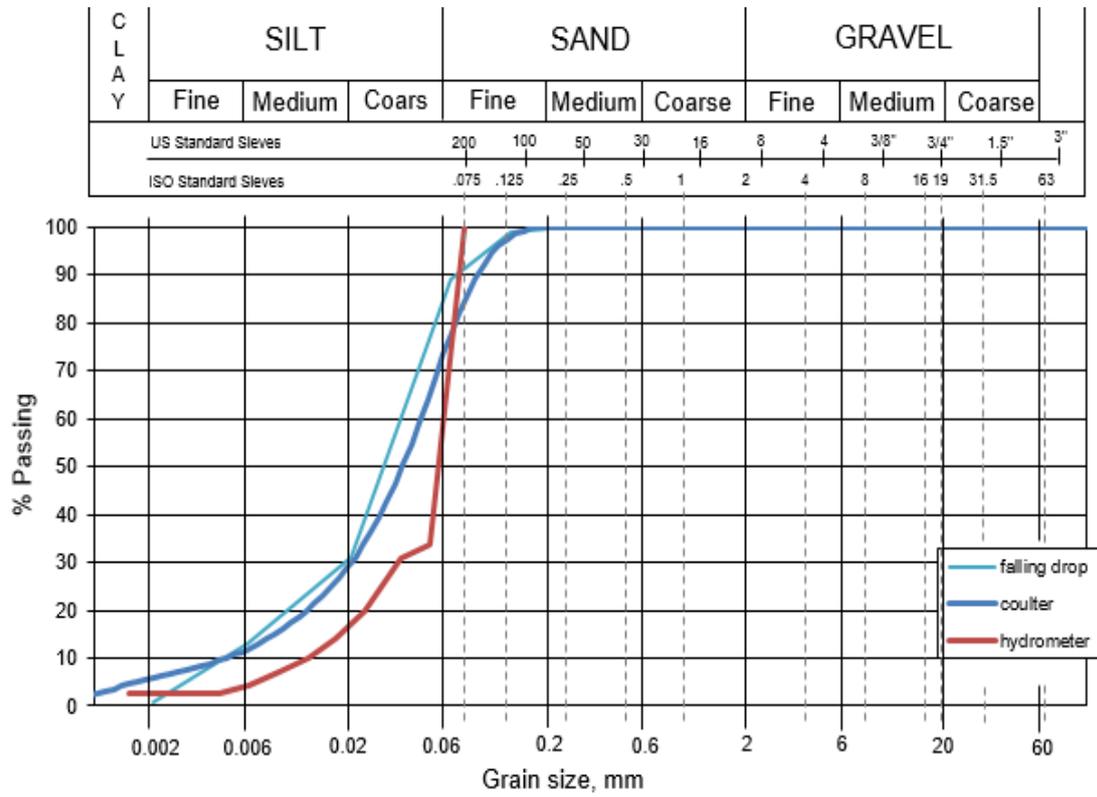


Hydrometer: Uniformity coefficient $C_u = d_{60}/d_{10} = 5$

Falling drop : Uniformity coefficient: $C_u = d_{60}/d_{10} = 10.5$

Coulter: Uniformity coefficient: $C_u = d_{60}/d_{10} = 7$

Cylinder 4 : 6-6,8 m

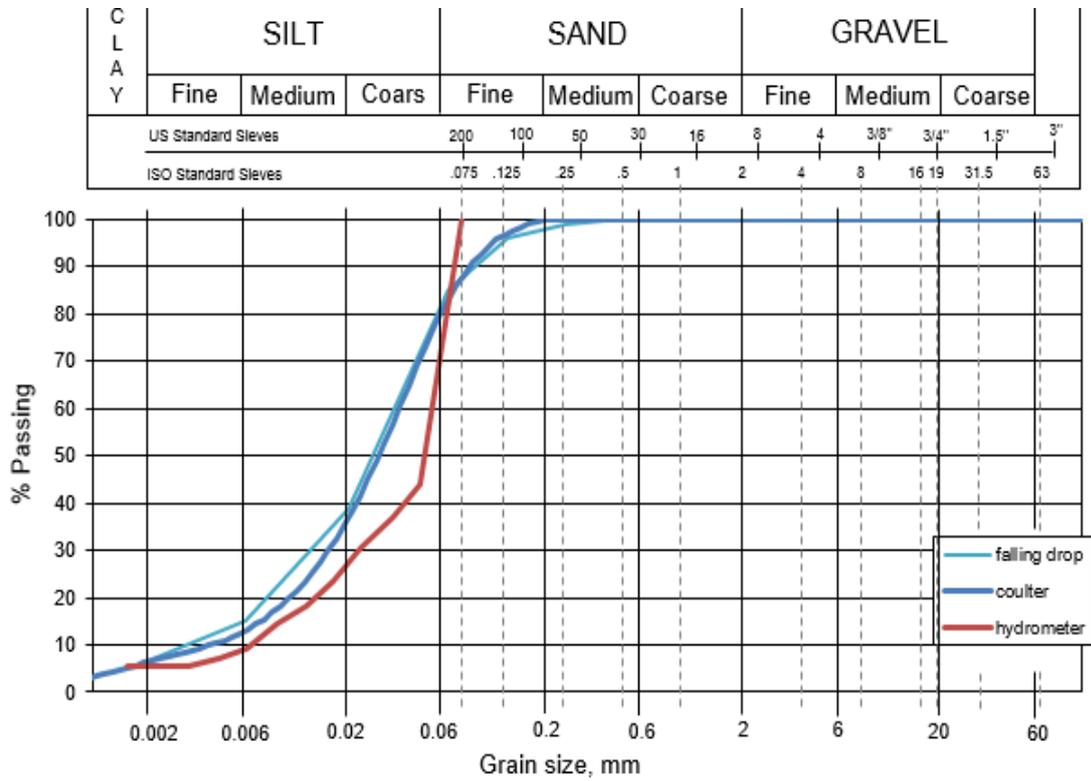


Hydrometer: Uniformity coefficient: $C_u = d_{60}/d_{10} = 6$

Falling drop: Uniformity coefficient: $C_u = d_{60}/d_{10} = 7.9$

Coulter: Uniformity coefficient: $C_u = d_{60}/d_{10} = 10$

Cylinder 11: 13-13,8 m



Hydrometer: Uniformity coefficient: $C_u = d_{60}/d_{10} = 9$

Falling drop: Uniformity coefficient: $C_u = d_{60}/d_{10} = 11.4$

Coulter: Uniformity coefficient: $C_u = d_{60}/d_{10} = 14$

3.2 Discussion

Cylinder 1

Description: Sand, silty, clayey, loose to medium dense. Well graded ($C_u > 6$).

- Agreement between falling drop and coulter method is good.
- Hydrometer results depart from the other tests, producing finer distribution. We observe an overestimation of the fine size fraction due to the particle-shape effect.
- Falling drop underestimates the ratio of the clay fraction.
- The ratio between the two methods and the hydrometer is 2/1 (if we multiply the results with 2 we get a very good correspondence). This is only for the coarser part of the chart because afterwards we have a good correspondence of the three methods.

Cylinder 4

Description: Silt, sandy, clayey. Well graded ($C_u > 6$)

- Agreement between falling drop and coulter method is good.
- Hydrometer results depart at some points from the other tests.
- Falling drop underestimates the ratio of the clay fraction.
- The ratio between the two methods and the hydrometer is $\frac{1}{2}$ (if we multiply the results with 0.5 we get a very good correspondence).

Cylinder 11

Description : Silt , clayey. Well graded ($C_u > 6$).

- Agreement between falling drop and coulter method is good.
- Hydrometer results agree with the other methods except from the silt where we have a less fine distribution.
- Falling drop underestimates the ratio of the clay fraction.
- The ratio between the two methods and the hydrometer is $\frac{1}{2}$ in the points with difference between the results (if we multiply the results with 0.5 we get a very good correspondence).

3.3 Conclusion

Overall, the study showed that there was no significant difference in the particle size distribution between Coulter and falling drop Method.

The falling drop systematically underestimates the ratio of the clay fraction while the coulter method didn't present any underestimation.

For coarser samples (cylinder 1) hydrometer overestimates the fine size fraction, while for finer samples (cylinder 4 and 11) we observe an opposite behavior. The reason may be the particle shape effect. The higher variation between the results is obvious in finer samples.

Starting from these results, more in-depth studies are needed. Above all it is necessary to verify how factor such as density and shape can influence the particle size analysis. More samples of various particle sizes are need to be tested for an accurate outcome.

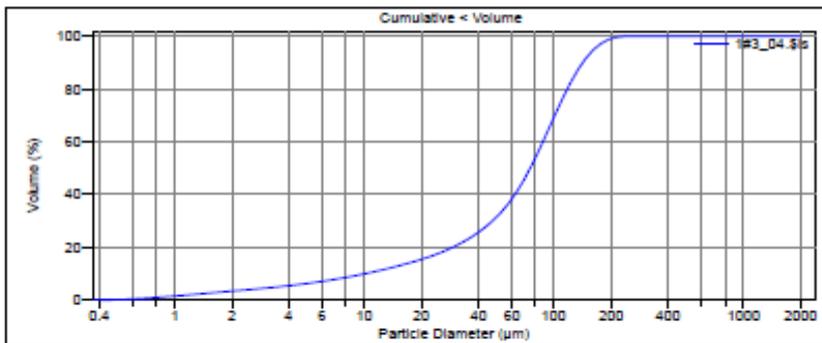
Appendix

Laser diffraction results (NGU)

Beckman Coulter LS Particle Size Analyzer

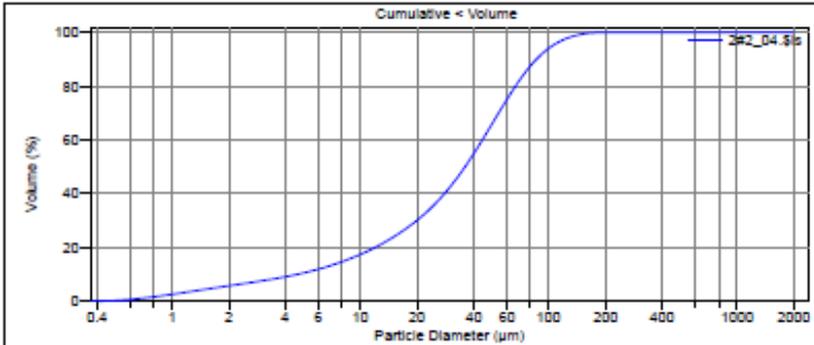
Page 1
19 Nov 2018 11:52

File name:	C:\LS13320\Rawdata LS 13320 Analyse\2018\Uten PID\20180217\1#3_04.\$ls		
File ID:	1#3_04.\$ls		
Sample ID:	2018.0217_3-3.8 m (Cylinder 1)		
Operator:	MSH		
Run number:	4		
Comment 1:	0,504g, ultrahyd		
Comment 2:	Fraunhofer		
Optical model:	Fraunhofer.r780d		
Residual:	0.29%		
LS 13 320	Aqueous Liquid Module	Run length:	60 seconds
Start time:	11:48 19 Nov 2018		
Pump speed:	45		
Obscuration:	9%		
Fluid:	Water		
Software:	6.01	Firmware:	4.00



Volume Statistics (Arithmetic)		1#3_04.\$ls	
Calculations from 0.375 µm to 2000 µm			
Volume:	100%	S.D.:	48.65 µm
Mean:	76.76 µm	Variance:	2367 µm ²
Median:	75.11 µm	C.V.:	63.4%
D(3,2):	15.03 µm	Skewness:	0.388 Right skewed
Mean/Median ratio:	1.022	Kurtosis:	-0.308 Platykurtic
Mode:	96.50 µm		
Specific Surf. Area:	3991 cm ² /mL		
dx: 10.08 µm	dx: 75.11 µm	dx: 142.4 µm	
<10%	<25%	<50%	<75%
10.08 µm	38.78 µm	75.11 µm	109.0 µm
			142.4 µm

File name: C:\LS\13320\Rawdata LS 13320 Analyse\2018\Uten PID\20180217\2#2_04.\$is
 File ID: 2#2_04.\$is
 Sample ID: 2018.0217_6-6.8 m (Cylinder 4)
 Operator: MSH
 Run number: 4
 Comment 1: 0,272g, ultralyd
 Comment 2: Fraunhofer
 Optical model: Fraunhofer,r780d
 Residual: 0.15%
 LS 13 320 Aqueous Liquid Module
 Start time: 12:18 19 Nov 2018 Run length: 71 seconds
 Pump speed: 45
 Obscuration: 9%
 Fluid: Water
 Software: 6.01 Firmware: 4.00

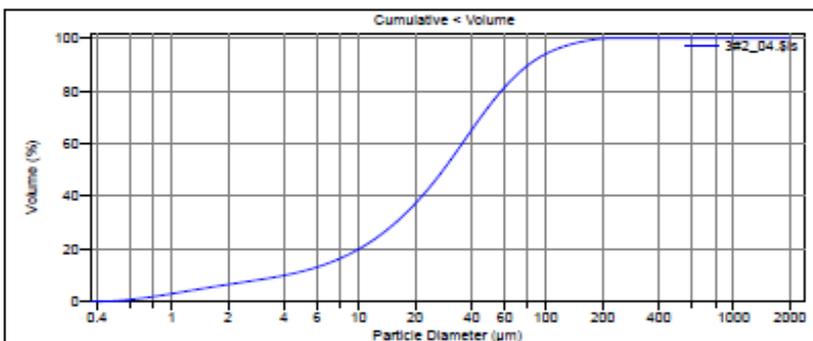


Volume Statistics (Arithmetic) 2#2_04.\$is
 Calculations from 0.375 μm to 2000 μm

Volume:	100%	S.D.:	32.51 μm
Mean:	41.71 μm	Variance:	1057 μm^2
Median:	35.96 μm	C.V.:	77.9%
D(3,2):	9.065 μm	Skewness:	1.038 Right skewed
Mean/Median ratio:	1.160	Kurtosis:	1.116 Leptokurtic
Mode:	50.23 μm		
Specific Surf. Area:	6619 cm^2/mL		

d10:	4.617 μm	d50:	35.96 μm	d90:	86.13 μm
<10%	4.617 μm	<25%	15.88 μm	<50%	35.96 μm
		<75%	59.99 μm	<90%	86.13 μm

File name: C:\LS13320\Rawdata LS 13320 Analyse\2018\Uten PID\20180217\3#2_04.\$is
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 Operator: MSH
 Run number: 4
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 Comment 2: Fraunhofer
 Optical model: Fraunhofer.r780d
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 LS 13 320: Aqueous Liquid Module
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 Obscuration: 10%
 Fluid: Water
 Software: 6.01 Firmware: 4.00

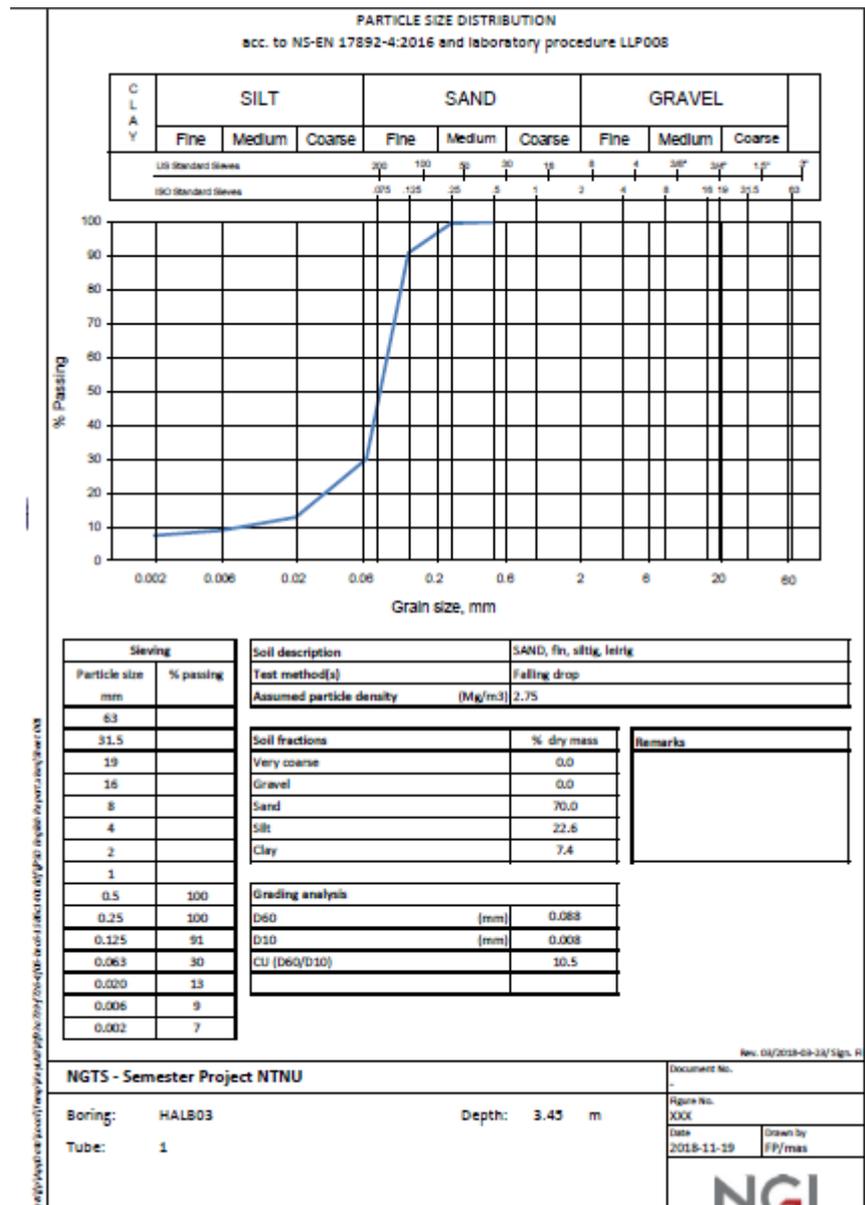


Volume Statistics (Arithmetic) 3#2_04.\$is
 Calculations from 0.375 µm to 2000 µm

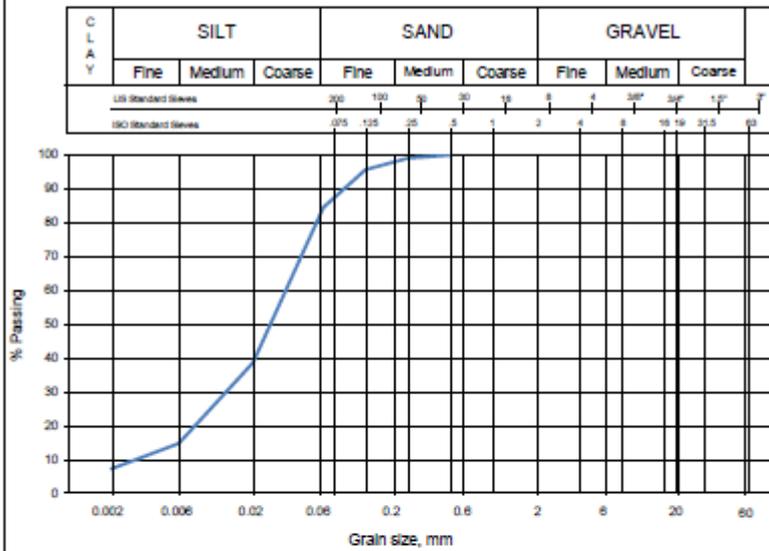
Volume:	100%	S.D.:	34.18 µm
Mean:	37.17 µm	Variance:	1168 µm ²
Median:	28.27 µm	C.V.:	92.0%
D[3,2]:	8.006 µm	Skewness:	1.750 Right skewed
Mean/Median ratio:	1.315	Kurtosis:	3.922 Leptokurtic
Mode:	37.97 µm		
Specific Surf. Area:	7494 cm ² /mL		

d10:	3.988 µm	d50:	28.27 µm	d90:	81.04 µm
<10%	<25%	<50%	<75%	<90%	
3.988 µm	12.85 µm	28.27 µm	50.64 µm	81.04 µm	

Falling drop results (NGI)



PARTICLE SIZE DISTRIBUTION
acc. to IS-EN 17892-4:2016 and laboratory procedure LLP008



Sieve		Soil description	SILT, leq
Particle size mm	% passing	Test method(s)	Falling drop
53		Assumed particle density (Mg/m ³)	2.75
31.5		Soil fractions	% dry mass
19		Very coarse	0.0
16		Gravel	0.0
8		Sand	15.5
4		Silt	77.3
2		Clay	7.2
1			
0.5	100	Grading analysis	
0.25	99	D60 [mm]	0.034
0.125	96	D10 [mm]	0.003
0.063	85	CU (D60/D10)	11.4
0.020	39		
0.006	15		
0.002	7		

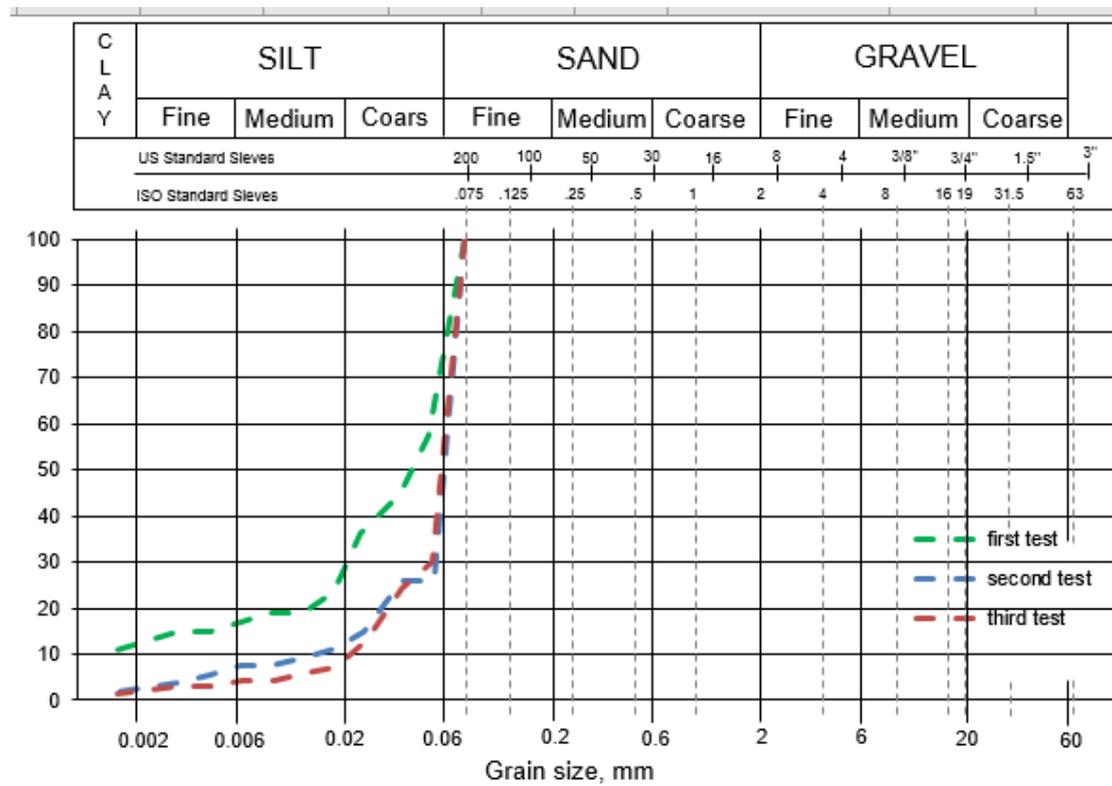
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NGTS - Semester Project NTNU		Document No.	-
Boring: HALB03	Depth: 13.40 m	Figure No.	XXX
Tube: 11		Date	2018-11-19
		Drawn by	FP/mas

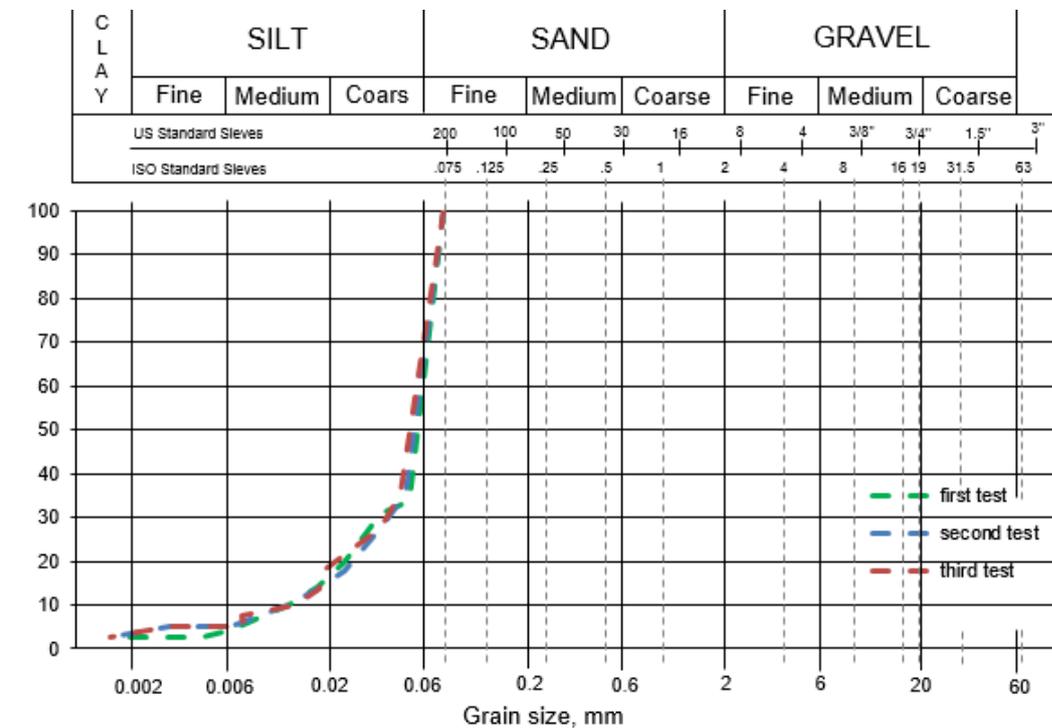


Hydrometer results (NTNU)

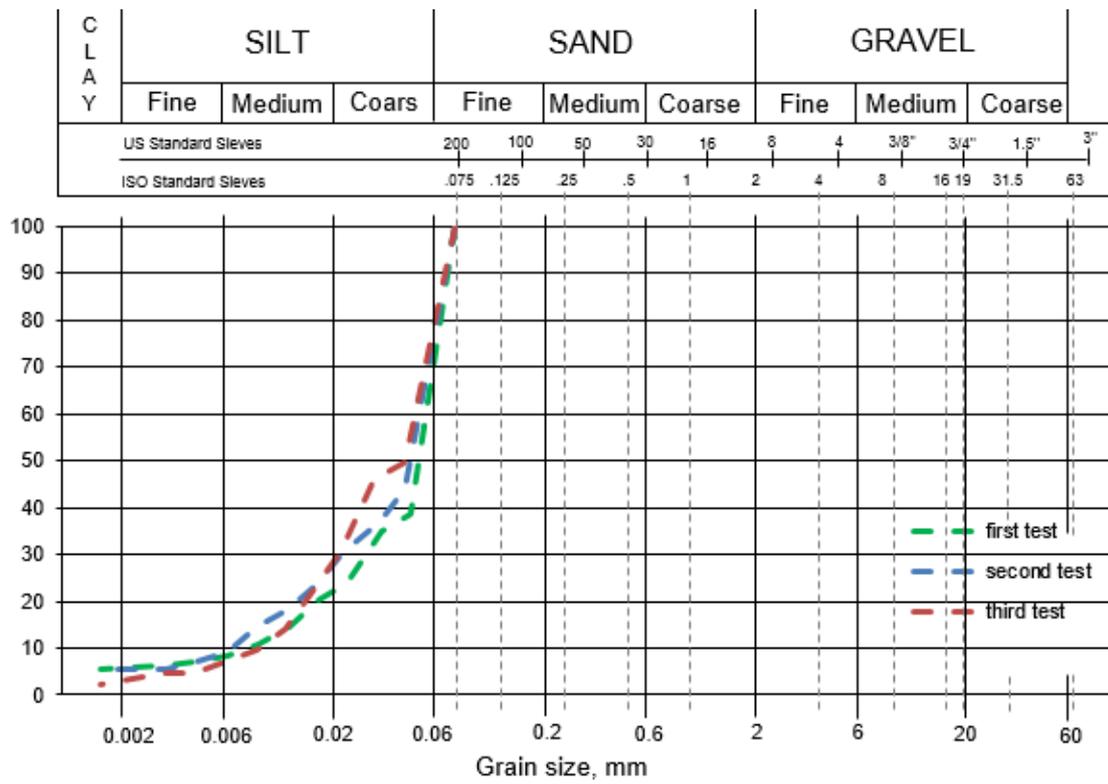
Cylinder 1:



Cylinder 4:



Cylinder 11 :



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