

Determination of the particle size distribution with the SEDIMAT 4-12

Training in Methodology and Device



2016 | Jürgen Kler | Soil Physics



Content

1. Prolog
2. Determination of the particle size distribution
3. Sampling and sample preparation
 - 3.1. Preparation of documentation and instruments for sampling
 - 3.2. Sampling in the field for the SEDIMAT 4-12
 - 3.3. Transport of the samples
 - 3.4. Sample preparation for the analysis
4. Steps for the determination of the particle size distribution
5. Creation of a test report
6. Physical background of the sedimentation analysis
7. Literature



1. Prolog

The particle size composition (texture) of soils is an essential factor for the evaluation of the soil fertility. The classification of the soil types according to the particle size composition is based on the different properties of the individual particle fractions. The sand fraction, together with coarser components, form the framework of the soil (soil skeleton), which gives it resistance to erosion and prevents the silt and clay particles from sticking together. It causes the favorable physical properties of the soil, like good ventilation and a high water mobility. On the other hand, the sand has little ability to trap water and nutrients, and favors high levels of leachate, soil permeation and shifting.

As the proportion of clay and silt increases, the water-holding capacity of the soil increases, but the mobility of water and the aeration decrease. The filter effect of the soil increases considerably, because as a result of the narrow pores between the soil particles slurried soil particles are usually mechanically retained, so that the leachate drains clear.

Depending on the particle size composition, the soils have the most diverse characteristics. Clay soils generally have favorable chemical but very poor physical properties. Although the nutrient content and the sorption capacity of these soils are usually very high, other properties that are equally important for plant growth, such as water transport and aeration, are very unfavorable in clay soils. Due to the low ventilation microorganisms and root respiration are constrained, so that the soil is biologically inert. The high clay content makes them difficult to work with, thus they are referred to as heavy soils, so they can often be used only as meadows and pastures. On the other hand, although the sandy soils show good aeration and water permeability, but due to the lack of sorption carriers, a lower water and nutrient retention capacity. The leaching losses of plant nutrients are therefore very high. The soils are generally very biological active, heat up quickly and are easy to work with (light soils).

Extremely clay-poor sandy soils mostly bear forest and heath vegetation and are often suitable for arable crops only with artificial irrigation.

The most fertile farmlands are in the middle of these extreme soil types. Accordingly, the most fertile and productive soils are the soils with about 30-40% sand and 60-70% silt and clay. The determination of the soil type since Albert Thaer forms a valuable basis for the assessment of the fertility state of a soil and is also used in the soil assessment carried out in Germany.

Specifying the type of soil alone is not sufficient to characterize soil fertility, since soils of the same soil type have a different fertility state. It must therefore be considered to characterize the fertility of a soil and other soil properties such as the mineral composition, humus, structure and nutrient status, and chemical and biological properties.



In summary, the soil type is an extremely important feature for the derivation of ecological soil properties. It determines based on its particle size distribution together with its primary structure a part of the pore volume and thus the water storage capacity and water conductivity. The particle surface determines the nutrient and pollutant storage capacity and the proportion of silt and fine sand the erosion susceptibility of the uncovered soil. The table below summarizes the different properties of the soil types.

Properties of the soil types

Property/Soil type	Sand	Silt	Clay	Loam
Treatment	++	±	--	+
Nutrient storage	--	-	++	+
Nutrient availability	-	+	+	++
Pollutant accumulation	-	+	++	++
Water holding capacity	--	+	++	++
Water availability	-	++	-	+
Mechanical filtering	+	++	-	+
Physico-chemical filtering	--	-	++	+
Drainage	++	--	-	±
Erodibility	±	+	--	-

Legend to the table

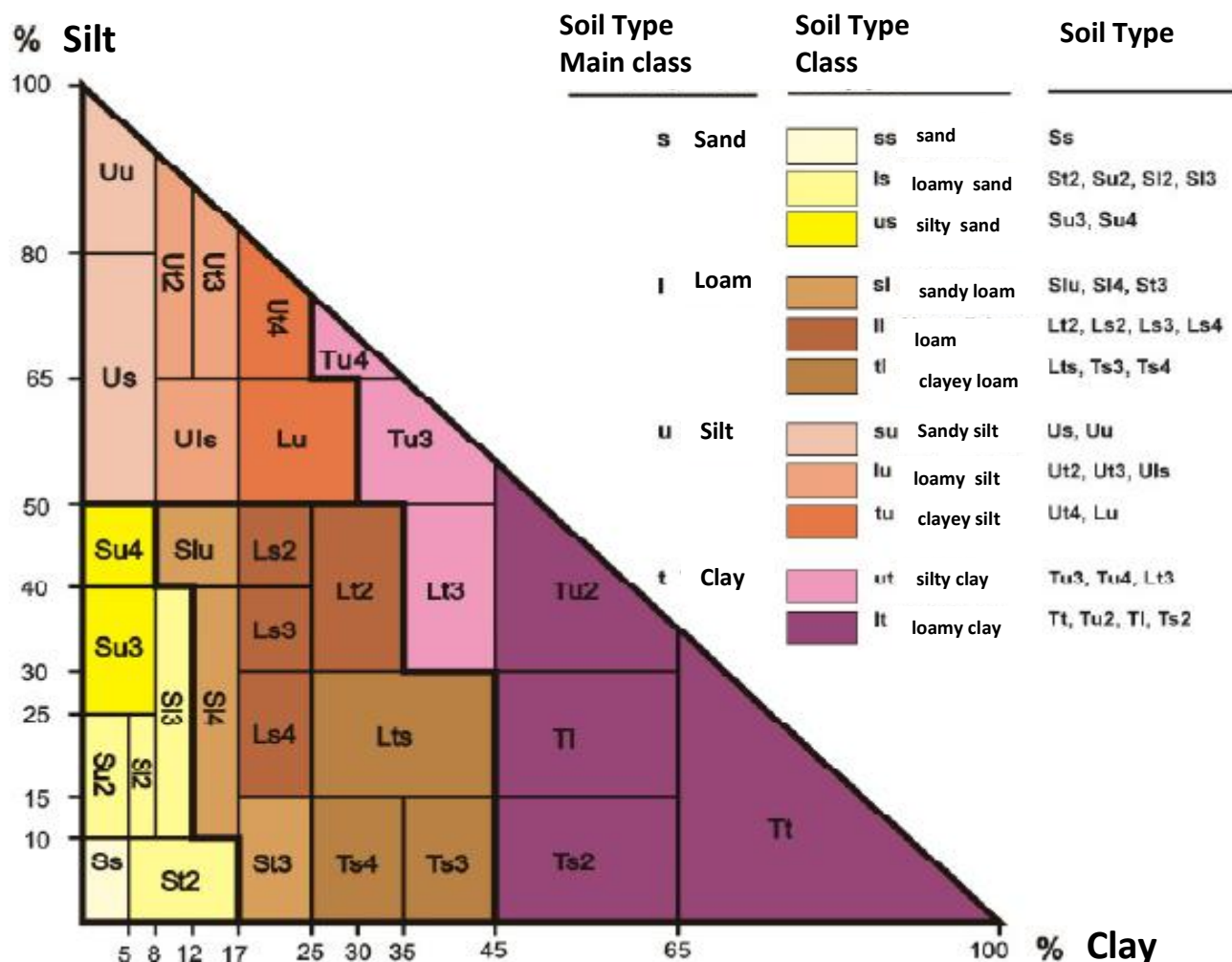
Signature	Meaning
++	very good (very high)
+	good (high)
±	sufficient (medium)
-	poor (low)
--	very poor (very low)

The determination of the particle size distribution is of great importance not only for agriculture and forestry, but also for institutions looking for suitable land for the construction of a building. This also includes authorities such as the road construction office, tax office and district office, which need a soil assessment for their respective concerns, and also often individual landowners.

The soil type triangle

If no further details are given, the soil type refers to the fine soil (equivalent particle diameter <2 mm). The proportion of coarse soil is given only as additional information. In the case of a share of >75% of the coarse soil on the total soil, the fraction of the coarse soil is given instead of the soil type. The fine soil is divided into the soil types sand, silt, and clay which can be represented in the soil type triangle. Loams contain all three fractions in similar proportions.

Each soil can be mapped according to its composition as a point in the soil type triangle. The colored areas indicate the affiliation of the soil to a soil type corresponding to the main fraction. If a fraction dominates less strongly, the second strongest fraction is indicated by the addition sandy, silty, clayey or loamy and represented by additional lower case letters.



2. Determination of the particle size distribution

The results of the particle size distribution serve for the exact determination of the soil type as well as for the estimation of the storage and exchange capacity for water, nutrients and pollutants, the aeration, workability and drivability of soils. The determination of the particle size distribution is carried out in two steps. A wet sieving separates the fine particles (particle diameter <0.063 mm) from the sample. The mass fractions of the total sample of the particle sizes in the sieve residue are determined by means of a dry sieving with test sieves, while the sedimentation analysis with the **SEDIMAT** is carried out for the determination of the fine particle fractions in the sieve pass. In the following, the individual steps for the determination of the particle size distribution from the sampling to the application of the **SEDIMAT** are explained. The method is suitable for determining the mass fractions of mineral particle size fractions in mineral soils of all kinds with organic matter contents of up to 10%.

The texture analysis procedure is based on the **DIN ISO 11277** standard and is applicable to mineral soils and the mineral content of organic soils. After appropriate pretreatment also on salt and carbonate soils.

The following devices are needed to determine the particle size distribution:

Water bath

- Weigh 10 g of air-dried soil in a centrifuge beaker
- Destruction of the organic substance with 30% by weight Hydrogen peroxide (H_2O_2) solution



Overhead shaker

- Disperse after removal of soluble salts and gypsum



Wet sieving

- Wet sieving through sieve with 0.063 mm mesh size
- dry and weigh on the sieve remaining sand



SEDIMAT 4-12

- Sedimentation with the SEDIMAT 4-12
- Dry sedimentation trays with contents up to constant weight



3. Sampling and sample preparation

3.1. Preparation of documentation and instruments for sampling

Preparation of the field notes

Field notes are necessary to accurately document the circumstances on the test field. They contain many important data (e.g., the location, the name of the experiment, the nature of the experiment, date, etc.). The documentary will later be transmitted in digital form, in order to subsequently determine relationships for analysis.

The proposed field note example is suitable for agricultural land.

Every other area e.g. (Road construction, civil engineering, building construction, etc.) may require a different field note, depending on what data is required.

Instruments for sampling

Sampling Rings

The picture shows a sample ring with a volume of 250 cm³. The wall thickness is about 2 mm. The lower edge is sharpened from the outside (cutting edge). The sample ring has an engraved number for exact assignment of the removed sample. The weight of the sample ring can be determined by weighing before sampling. This may be useful if the weight of the sampled soil is of interest and the sample should not be destroyed during weighing. The sample ring is closed with two lids.



Sampling Instruments

The picture shows the instruments needed to take soil samples.



- 1 Folding rule or tape measure
- 2 Sample ring
- 3 Driving cap
- 4 Hammer
- 5 Spatula, spade
- 6 Knife, stainless and sharp
- 7 Lids

3.2 Sampling in the field for the SEDIMAT 4-12

The following describes the removal of undisturbed soil samples. This method is applicable to stone-free and low-grade stony soils. The removal of the soil core samples of known volume is carried out with the shown thin-walled metal sample rings (100 cm³ or 250 cm³, each ring carries its own number and has its own weight, which is important for the calculation).

The sample ring has to be pressed or hammered sufficiently deep into the soil without bending and without compaction of the soil, either in a vertical or horizontal ground surface, until the sample ring is filled slightly beyond the upper edge.



For preservation of the natural structure, carefully pull the sample ring out. To do this, remove the soil protruding from the ends of the sample ring with a sharp knife or a sharp spatula. The volume of the soil samples thus corresponds to the volume of the sample ring. At least six soil core samples shall be taken from each soil layer.



The sample rings have to be cleaned from the excess soil, sealed with plastic lids and the number has to be entered in the field notes. Then store them in a stable box for transport.

3.3 Transport of the samples

The undisturbed soil samples are taken after removal of the rings from the ground, smoothing the surfaces and capping with lids to protect against loss of water by evaporation in tightly sealable transport containers and brought to the laboratory.

3.4 Sample preparation for the analysis

In preparation for the analysis, the soil sample to be examined is air-dried or in the laboratory in the oven at a maximum of 40 ° C and, if necessary, mechanically coarsely crushed. Subsequently, the whole soil sample is forced through a sieve with a mesh size of 2 mm to separate the coarse soil from the fine soil. At least 100 g of fine soil should then be available for a soil sample.

Transfer the fine soil completely into a sample divider and divide the sample mass until the mass of the subsamples is approx. 10 g. For a meaningful study, at least three parallel subsamples of a soil sample must be generated. However, for the analysis with the SEDIMAT, the maximum number of samples, which is 13 samples (12 test samples + 1 sample for determining dry matter content) should be used as much as possible in order to ensure the maximum informative value and effectiveness.

The dry matter content is determined on one of the subsamples. This one cannot be used for further investigation. The remaining parallel samples are weighed with the precision balance and the dry matter is calculated on the basis of the calculated dry matter fraction.

Depending on the composition of the soil sample further preparation steps are necessary. If the soil contains more than 2% of organic matter, it should be destroyed. For soluble salts at a concentration greater than 0.5%, their removal is recommended.



4. Steps for the determination of the particle size distribution

4.1 Air drying the sample

Screening of the sample, which has passed through the 2 mm sieve, is released air-dry for analysis.

4.2 Destruction of organic matter

The destruction of the organic substance is carried out with about 30 ml of 30 weight-% hydrogen peroxide (H_2O_2)-solution.

10 g of the soil sample are wetted in a centrifuge beaker with 30 ml H_2O , placed on a water bath. Add a few drops of H_2O_2 and stir carefully. In most cases, a violent reaction begins that lasts longer (about 1/2 to 1 h). The sample is allowed to stand on the water bath until the reaction subsides. Then add a little H_2O_2 again and stir. The process is repeated until it is visible that the reaction no longer causes violent foaming. Now then, residual hydrogen peroxide can be added. For complete humus destruction, the samples remain on the heated (about 95 °C) water bath for about 6 hours; after the heater is switched off, the samples remain on the water bath overnight.



4.3 Removal of soluble salts and gypsum

Step 1

If the electrical conductivity is $<400 \mu\text{S}/\text{cm}$, the sample is suitable for dispersion.

Step 2

If the electrical conductivity is $> 400 \mu\text{S}/\text{cm}$, the sample is filled up to 150 g total weight with H_2O , centrifuged, decanted, then replenished with H_2O to 150 g total weight, homogenized, shaken for 1 h, centrifuged and the electrical conductivity measured. If the electrical conductivity is $<400 \mu\text{S}$, the sample is suitable for dispersion. If not then repeat step 2.



4.4 Dispersion

Sufficient water is added to the centrifuge cup so that the total volume is about 200 ml. Add 25 ml of $\text{Na}_4\text{P}_2\text{O}_7$ (tetra-sodium diphosphate decahydrate) to the solution and shake it until all the soil is in suspension. Shake the centrifuge beaker in the overhead shaker for 18 hours.



4.5 Wet sieving with the 0,063 mm sieve

During the wet sieving, the sample suspension is washed through a sieve with a mesh size of 0.063 mm. The amount of water used should not be more than 1000 ml. Here it is important that the sieve passage is completely collected. The wet sieving can therefore be performed as shown in the picture. Using the spray bottle, water is added to the sieve and the sample is washed through the sieve. The sieve passage is completely collected in the stand cylinder. For easier handling, it is also possible to deviate from this design as long as the complete passage through the sieve is collected.



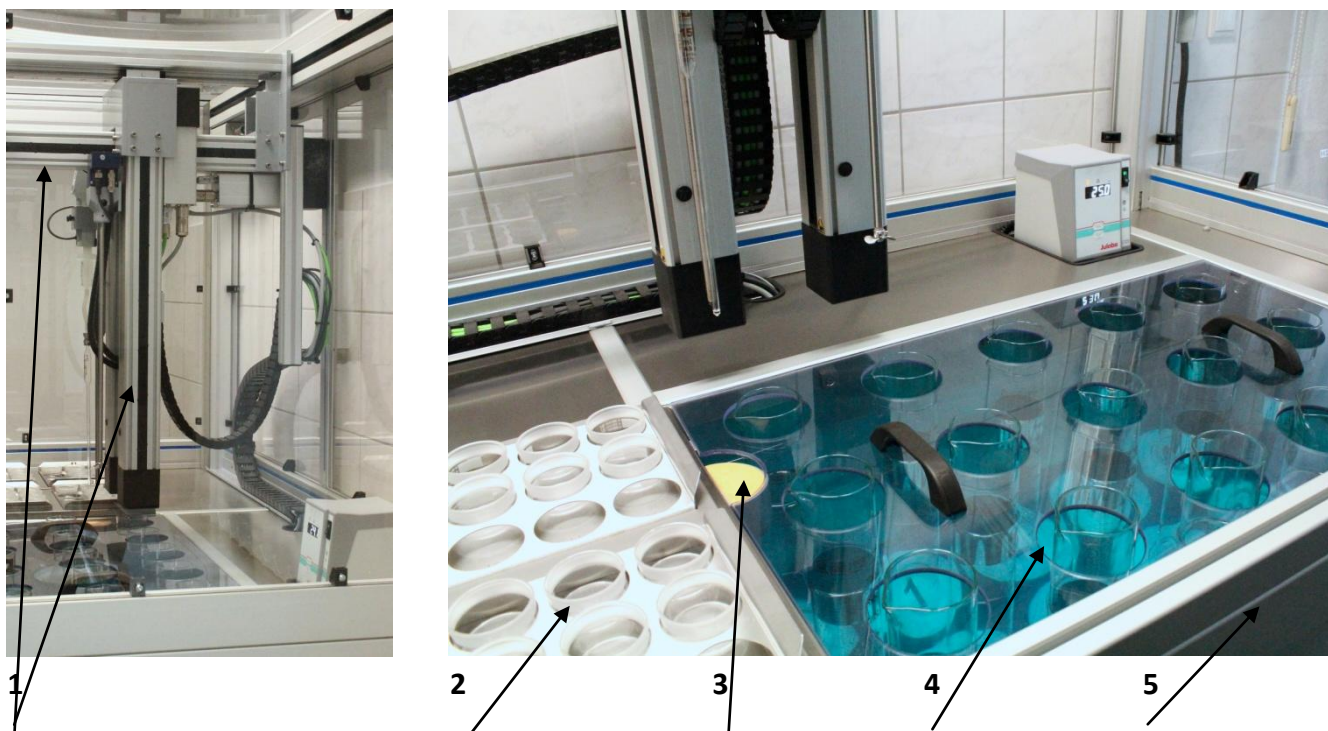
The sieve is taken out of the funnel and the residue in the sieve is rinsed by light spraying from the spray bottle into an evaporating dish. The tray is placed in a drying oven at a temperature between 105 ° C and 110 ° C until the residue (sand portion) is dry.

All particles adhering to the inside of the funnel are rinsed into the sedimentation cylinder. The dry residue (sand content) is cooled and sieved on sieves with opening widths <2 mm to 0.063 mm (dry sieving). The residue fractions on each sieve are 0.63 mm (coarse sand content), 0.20 mm (mean sand content), 0.10 mm (fine sand content), > 0.10 mm (fine sand content); are weighed and their masses logged. The suspension in the sedimentation cylinder is filled up to 1000 ml with water.

4.6 Sedimentation analyses of the fine particles

The analysis of the fine particle content in the sieve pass is automated with the SEDIMAT 4-12 (laboratory automat for the determination of the particle size fractions of the mineral soil substance according to DIN ISO 11 277 on 12 samples with 4 fractions). The components of the SEDIMAT are shown in the following figure.

Laboratory Machine



1 base frame for mounting linear axes; 2 magazines for the pipette samples;
3 cleaning station for stirrer and pipette; 4 tempered water bath; 5 safety cover and housing

Switch on the SEDIMAT. When the power supply is switched on, the SEDIMAT starts to heat the water bath to the set temperature. For the test procedure according to DIN ISO 11277 this is 25 ° C. Immerse the sedimentation cylinder (measuring cylinder) in the tempered water bath up to the 1000 ml mark. The measuring cylinder and its contents must be able to assume the temperature of the tempering tank (+ 25 ° C). If not all 12 cylinder spots are used, the measuring cylinders must be used continuously in accordance with the numbering, starting with location No. 1. The content of the measuring cylinder (suspension) is stirred with the stirrer, so that no sample remains on the ground. Once stirring is stopped, the program for sedimentation times is started.

Sedimentation times for the fractions- F1; F2; F3; F4

F1-	49 s
F2-	04:07 min
F3-	45:52 min
F4-	06:52:50 h

After expiry of these times, one sample each is taken with the pipette.



The pipette is lowered in the middle of the measuring cylinder until its tip has reached the appropriate depth below the surface of the suspension. The lifting piston of the pipettes moves upwards, so that the pipette is filled to 10ml with suspension. The pipette is pulled out of suspension and the contents are automatically stored in the desired Petri dish. The adhering to the inner walls of the pipette suspension is rinsed with water from the rinsing bath in the same Petri dish.

(For the desired fractions (F1-F4), the corresponding magazines are to be equipped with clean and balanced Petri dishes according to the installed measuring cylinders (soil samples).)

After the experiment, the Petri dishes are set in the oven at a temperature between 105 ° C and 110 ° C and evaporated to dryness. Thereafter, the Petri dishes are cooled in a desiccator and then weighed with the contents to 0.0001 g and determines the mass of the residue to 0.0001 g. If necessary, further samples are taken at the times indicated and according to the same procedure as above.

4.7 Calculation of the results for the fractions <2 mm

The calculation method assumes that the sample mass is the sum of the fractions and not the mass of the sample.

The mass [in g] of particulate matter in the 1000 ml suspension (m_{f1} , m_{f2} , m_{f3} , etc.) for each time of sampling with the pipette is calculated according to the following equation:

$$m_{fx} = m_{sx} (1000 / V_c)$$

Herein mean:

m_{fx} = Mass of suspended solids in 1000 ml suspension, in g;

m_{sx} = Mass of the material during the nth-sampling with the pipette;

V_c = Calibrated volume of the pipette, in ml.

The volume of the pipette is 10 ml as standard. The actually aspirated volume can be checked with the "additional program for volume determination of the pipette".

After starting the program, enter the "110" for "volume determination of the pipette" in the program selection dialog. In this program, 10 ml of water are removed from the rinse water cylinder P1 and stored in the Petri dish of fraction 1. The actual pipetted volume can be determined by weighing.



Analogously, the mass of the dispersant in the pipetted fraction is determined (blind value). The mass of the dispersant must be known since it must be subtracted from the obtained mass of the pipetted fractions.

Assuming a very accurate preparation of the sodium diphosphate solution, one can set the theoretical blind value of 0.0067 g for a volume of 10 ml of the dilute solution.

Alternatively, this value can be determined by adding 25 ml of the dispersant to a KÖHN cylinder and filling it up to 1000 ml with water. Mix the solution well, then let it stand for 1 h. Take at least three times 10 ml, transfer to Petri dishes or beakers (not aluminum vessels), dry as well as the soil samples, weigh, and form and note the mean value of the weighing results.

The solid weight in the 1000 ml of the dispersant solution, m_d , in g, is given by:

$$m_d = m_r (1000 / V_c)$$

Herein mean:

m_r = Mass of the particular residue, in g;

V_c = Calibrated volume of the pipette, in ml.

In the example, the masses of the pipetted fractions are designated as follows:

Fraction < 0,063 mm = m_{f1} ;

Fraction < 0,020 mm = m_{f2} ;

Fraction < 0,002 mm = m_{f3} .

The samples first taken with the pipette contain not only the target particle fraction but also particles of the smaller particle fractions. Furthermore, each sample contains the same mass of dispersant m_r .

Therefore, the masses of the fractions (in g) result in:

Mass of the fraction

0,063 mm bis 0,020 mm = $m_{f1} - m_{f2} = m(0,063 \text{ mm})$;

Mass of the fraction

0,020 mm bis 0,002 mm = $m_{f2} - m_{f3} = m(0,020 \text{ mm})$;

Mass of the fraction

<0,002 mm = $m_{f3} - m_d = m(0,002 \text{ mm})$.

The principle is extended to the other sampled fractions.



Thus, the mass of the sample <2 mm is equal to the sum of the masses of the individual fractions from the dry sieving and sedimentation analysis. This total sample mass is named m_t (in g)

The proportion in each fraction <2 mm is calculated as follows:

proportion = Mass of the fraction / m_t

The results are presented in the form of a table in which the proportion of each size fraction is entered in two significant digits. The basis on which the results are presented, e.g. as a proportion of the material to be screened <2 mm or as a proportion of the total soil, must be clearly indicated.

5. Creation of a test report

In some facilities, it makes sense to prepare a test report for the follow-up of the samples, which should include the following information:

- a reference to the international standard used
- the date of the laboratory analysis
- the test methods used, indicating the parts of the standard used; for alternatives, follow the instructions given in the standard
- the results of the sieve and pipette analysis
- sufficient information to clearly identify the sample in the laboratory protocols
- the name of the person performing
- the name and address of the laboratory



6. Physical background of the sedimentation analysis

The sedimentation analysis after Köhn is based on the following consideration:

A solid particle moves while sinking in a liquid at a constant rate. For low speeds (up to about 10^{-2} cm/s) one uses the approximate formula of Stoke:

$$V = \frac{d^2 \cdot (\rho_K - \rho_{fl}) \cdot g}{18 \cdot n} \quad [\text{cm/s}]$$

Herein mean:

- V** = sedimentation velocity [cm/s]
- d** = particle diameter [cm]
- ρ_K** = particle density [g/cm^3]
- ρ_{fl}** = liquid density [g/cm^3]
- g** = gravitational acceleration [cm/s^2]
- n** = dynamic viscosity of the liquid [g/cm]

According to this formula, the sinking rate depends on the following quantities:

1. Diameter of the particle
 2. Specific weight of the particle – both are given by the test material
 3. Density of the liquid
 4. Viscosity of the liquid
3. and 4. change strongly with the temperature, which must not change during the experiment.



7. Literature

DIN ISO 11277. Bestimmung der Partikelgrößenverteilung in Mineralböden.
Beuth- Verlag Berlin 2002-08.

Bundesanstalt für Geowissenschaften und Rohstoffe 2005: Bodenkundliche Kartieranleitung.

Umwelt-Geräte-Technik GmbH 2003: Laborautomat zur Bestimmung der Partikelgrößenverteilung in Mineralböden nach DIN ISO 11277.

Lehrbuch für Landwirtschaftsschulen „Pflanzliche Erzeugung“ Bodenkundliche Grundlagen.
München 2006.