

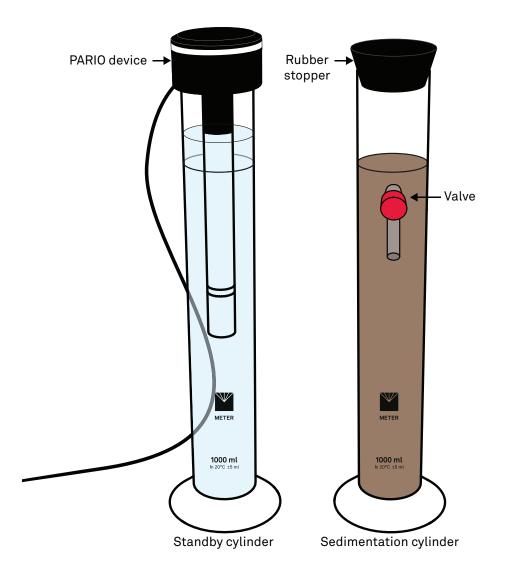
PARIO°

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1. INTRODUCTION

Thank you for choosing the PARIO Soil Particle Analyzer from METER Group. PARIO provides a more efficient way of measuring and grouping particles into separate ranges for weight and size. This manual includes information for two variants, PARIO Classic and PARIO Plus, or the PARIO system.

The PARIO system is based on the sedimentation of dispersed particles in suspension. The information covers the working steps in performing a reliable analysis, including guidelines for required and research-specific steps of soil sample preparation.

Detailed information about the PARIO Control software configuration, advanced settings, post processing, and evaluation is also included in this manual.

Verify all PARIO components are included and appear in good condition:

- PARIO device
- · Standby glass sedimentation glass cylinders
- Sedimentation glass cylinder with outlet
- Rubber stopper
- Beaker, glass (PARIO Plus)
- USB cable
- · Product support information card

2. OPERATION

Please read all instructions before operating the PARIO to ensure it performs to its full potential. Do not apply any other PARIO installation or measurement procedures other than those described in this manual.

Please observe the following considerations carefully.

- Read all applicable safety instructions (e.g., material safety data sheets for used chemicals or soil preparation instructions) and follow recommended safety measures.
- Do not use the device if the electrical wire is damaged.
- Always avoid direct sunlight exposure.
- Dispose of all materials and chemicals according to national legislation and environmental care regulations. Please refer to the respective safety data sheets.



PRECAUTIONS

METER instruments are built to the highest standards, but misuse, improper protection, or improper installation may damage the sensor and possibly void the manufacturer's warranty. Before integrating PARIO into a system, make sure to follow the recommended installation instructions and have the proper protections in place to safeguard sensors from damage.



IMPORTANT: Never touch the pressure sensor, which is shielded by the sensor cover. For cleaning, spray a soft water jet into the area of the sensor protection to avoid damaging the pressure sensor.

2.1 INSTALLATION

Follow the steps listed in Table 1 to set up the PARIO.

Table 1 Installation

Tools Needed	Microsoft® Windows® computer (Windows 10)
	WARNING: The PARIO is not intended for outdoor use.
Preparation	Select Clean, Level Location Place the PARIO on a horizontal, vibration-free, and solid surface where the temperature remains fairly stable (away from air conditioner and heater vents, windows, etc.).
	IMPORTANT: Temperature fluctuations or vibrations will strongly affect the accuracy of measurement results.

Table 1 Installation (continued)

	Install PARIO Control Software and METER USB Driver Download the installation package from metergroup.com/downloads/pario.
	Run the PARIO CONTROL *.exe file and go through the PARIO Control Setup Wizard. The wizard will install the PARIO Control software and the METER USB driver.
Installation	Click on the PARIO Control icon (located where saved in the wizard installation) to open the program (Section 2.4.2).
	Connect PARIO Connect the PARIO USB cable into a computer USB port. The PARIO LED should flash white three times and then turn off. (Section 2.4.2).
	Multiple PARIO devices can be used simultaneously by connecting them to a powered USB hub. METER recommends a maximum of 15 PARIO devices per computer.

2.2 KEY FACTORS TO GET RELIABLE MEASUREMENTS

Getting reliable results from PARIO measurements is dependent on five key factors. described in the next sections: accurate mass of oven-dry materil, accurate sand percentages, accurate mass of salt dispersion, temperature equilibration, and avoiding gas bubble formation during measurement.

2.2.1 ACCURATE MASS OF OVEN-DRY MATERIAL

PARIO derives the silt content from the pressure changes as absolute mass and relates this to the original mass. Thus, any error in the dry soil mass strongly influences the analysis. An incorrect total mass can be caused, for example, by using air-dry material and entering this in the PARIO Control interface as total (oven) dry mass. To avoid this error, the gravimetric water content in the air-dry mass must be known to recalculate the oven-dry mass. Another source of error can be caused by not considering the mass loss by destruction of organic matter during the preparation of the suspension. If organic matter is destroyed, it also must be subtracted from total air-dry mass (see below for a calculation example of net particle mass).

As a general rule, destroying organic matter is always a good idea, because organic matter binds clay particles. Also, practical experience with PARIO measurement has shown that results are better if organic matter is removed (Section 2.3).

EXAMPLE CALCULATION FOR NET PARTICLE MASS

$$m_{particles} = m_{dry} - m_{OM}$$

 $m_{dry} = m_{airdry} - m_{w}$

Here, $m_{particles}$ is the total oven-dry mass of particles (to be inserted in PARIO), m_{aindry} is the mass of airdry material used in the analysis, m_w is the mass of water contained in the air-dry material, and m_{OM} is the mass of organic matter in the oven-dry sample that has been destroyed in the process of sample preparation. The masses m_w and m_{OM} can be calculated separately by

$$m_w = \frac{m_{airdry}}{(1 + c_w)}$$

and

$$m_{OM} = m_{dry} \over (1 + c_{OM})$$

where c_w [g/g] is the gravimetric water content of the material used in the PARIO test, and c_{OM} [g/g] is the concentration of organic matter. Note that the concentration of organic substance c_{OM} can be estimated from organic carbon content c_{OC} [g/g] by assuming $c_{OM} = 1.72 c_{OC}$.

2.2.2 ACCURATE SAND PERCENTAGES

Especially in PARIO Classic mode, it is very important that the sand fractions obtained by screening are accurate and reliable. Since PARIO determines only the silt fractions from the pressure time series, the total cumulative particle distribution curve is obtained by extrapolation from the sand fractions toward the clay fraction. Thus, an error in the sand fractions will propagate toward the clay fraction. For soils with small clay content, this can cause a very large relative error of the clay fraction. This problem is relieved by PARIO Plus, because it gives a robust estimate of the clay fraction and in the case of a mass conflict between external sand fraction and measured clay fraction, the silt particle mass is scaled to give a total fit that fulfills the total mass balance. Minor errors in the entered sand fractions are less problematic in PARIO Plus mode.

2.2.3 ACCURATE MASS OF DISPERSION SALT

Especially in PARIO Plus mode, it is very important to know the exact concentration of dry dispersion salt in the suspension. This is because the mass of sediment in the released suspension is calculated by subtracting the mass of dispersion salt from the total dry mass. To get the right result, it is crucial that this mass of dry salt (the salt error, known also from pipette analysis) is exact. The amount of dry salt in the suspension is not equal to the gross mass of the salt added to the suspension.

Many salts are hygroscopic or even delivered with a certain mass of water (e.g., tetra-sodium diphosphate decahydrate, $Na_2P_4O_7 \times 10~H_2O$). So, it is important to either know the exact amount of water the salt has and subtract this amount when entering the value through the

PARIO Control interface, or determine the salt error, as in the usual pipette analysis. In any case, the dry salt mass [g], which refers to the total suspension volume, must be used. Minor errors in the salt mass are less problematic in the PARIO Classic mode.

EXAMPLE CA	LCULATION FOR SALT MASS	
а	Type of dispersant	$Na_2P_407\times 10\; H_2O$
b	Dry mass of sample	25 g
c	Volume of dispersant solution that is added to the sample suspension	25 mL
d	Original concentration of dispersant salt in dispersant solution	44.6 g/L
e	dry concentration of dispersion salt in dispersion solution	26.6 g/L
$f = d \times b$	Mass of salt in dispersant solution that is added to the suspension	1.115 g
$g = e \times b$	Mass of dry salt in dispersant solution that is added to the sample solution	0.665 g
h = g	Mass of dry salt in total suspension	0.665 g

The value in field g and h, is to be entered into the Mass of dispersant field (Figure 16).

2.2.4 TEMPERATURE EQUILIBRATION

The measurements must not be distorted by temperature gradients that may be caused by radiation or cooling air flows. Therefore, the water in which the PARIO device is stored in the standby position and the test suspension must have exactly the same temperature. In practice, this is achieved by ensuring that all processing steps take place in the same room or, if the suspension is prepared in an adjacent room, that the temperature in the rooms is absolutely identical. In addition, the PARIO measurement must never be positioned near windows with sunlight or under air-conditioning systems. If the PARIO device is placed in a suspension with a different temperature, the PARIO shaft will expand or shrink slightly and this will affect the pressure readings, especially in the first few minutes until a temperature equalization between the PARIO device and the liquid has taken place.

2.2.5 AVOID GAS BUBBLE FORMATION DURING MEASUREMENT

Gas bubbles in a suspension might form due to warming or due to microbial activity in the sediment. Also, incomplete removal of H_2O_2 used in the oxidation of organic matter can cause a microscopic gas bubble formation. This might be seen by a kind of very fine foam on the surface of the suspension. Gas bubbles may distort the readings of the pressure transducer, if bubbles are directly attached to its surface. Gas bubbles that form in the sediment that accumulates at the base of the sedimentation cylinder will lead to increasing pressure readings, because the water column above the measuring depth is lifted by bubble expansion.

OPERATION

To avoid gas bubble formation, it is of advantage to use slightly degassed water (e.g., by heating the deionized water prior to its use in preparing the suspension). To avoid gas bubble formation by microbial activity under reducing conditions, as might occur in fertile soils, it is recommended that such materials are quickly processed after organic substance destruction and dispersion. To avoid gas bubble formation due to incompletely oxidized organic matter, ensure that all H_2O_2 is expelled by sufficient heating in the preparation process (Section 2.3).

2.3 SOIL SAMPLE PREPARATION

Users familiar with the hydrometer method or the pipette method and use the automated analysis by PARIO as a replacement for these techniques can prepare the soil sample suspensions the same way. They can continue with Section 2.4.

Sample preparation is of key importance for the results of the particle size analysis. In standard pipette and hydrometer procedures, and thus also for PARIO measurements, it involves the destruction of binding agents and the dispersion of the particles. Internationally, there are many varieties of sample preparation procedures in the sedimentation analysis, which differ in the specific steps and sequence of treatments.

The raw material that is used in the analysis should consist of air-dry particles with diameters < 2 mm. Complete de-agglomeration of particulate materials is a prerequisite for good results in PSA. The typical preparation steps to get the raw material is described in the following table.

Soil dispersion can be accomplished using a combination of chemical and mechanical means. Chemical dispersion uses hydrogen peroxide (H_2O_2) and sodium hexametaphosphate [HMP, $(NaPO_3)n$] (also known as sodium metaphosphate) or sodium tetraphosphate (also known as sodium pyrophosphate). Hydrogen peroxide oxidizes organic matter, which binds soil particles into aggregates. The function of HMP is to complex any $Ca2^+$ in solution and to replace $Ca2^+$ with Na^+ on the ion-exchange complex of soil particles, resulting in the dispersion of individual soil particles and causing break down of soil aggregates. Mechanical agitation used in conjunction with chemical treatments enhances the dispersion (Ketteler et al., 2001).

For accurate analysis, it is crucial to know exactly the dry mineral sample mass that is used in the analysis. Since air dry soil has still some water in it, the gravimetric water content of air-dry soil must be known to obtain the total dry (oven dry) mass. Furthermore, if other treatment steps lead to mass loss, such as organic matter removal, salt removal or removal of binding oxides, the corresponding mass loss must be quantified and considered in the analysis (Section 2.3.1).

METER thus recommends preparing two separate soil samples.

- one for the PARIO analysis (called "Measurement sample"), and
- one to determine the real mass loss from the raw material that is caused by the treatment (called "Pretest sample")

PARIO

Using the pretest sample method allows for the safe determination of the net dry mineral soil mass that actually settles in the sedimentation cylinder.

Figure 1 shows all recommended steps for the pretest and measurement sample preparation and the PARIO measurement. Air drying, sieving, destroying organic matter, and dispersing sample particles are necessary to get a valid result of particle size distribution (PSD) (Section 3.4). Figure 1 also includes optional pretest preparation steps of removing soluble salts and plasters and removing iron oxides. These optional steps depend on the user's needs and scope of research.

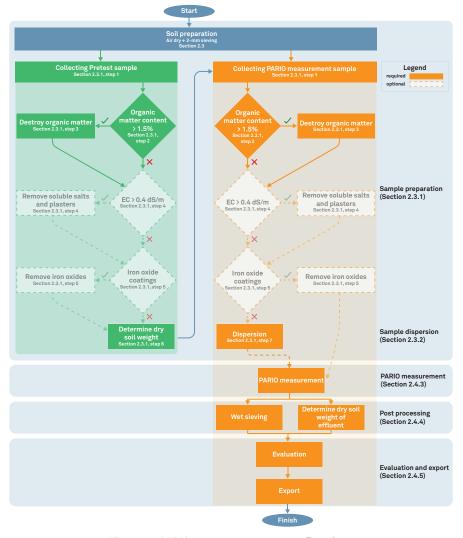


Figure 1 PARIO measurement process flowchart

OPERATION

For accurate analysis, it is crucial to know the exact dry mineral sample mass that is used in the analysis. Since air dry soil still has some water in it, the gravimetric water content of air-dry soil must be known to obtain the total oven-dry mass. Complete dispersion of soil is a prerequisite for good results in particle size analysis. Furthermore, if other treatment steps lead to mass loss, such as organic matter removal, salt removal, or removal of binding oxides, the corresponding mass loss must be quantified and considered in the analysis

Steps		Equi	pment Needed
-	and sieve the ground sample to gain approximately 150 g material.	•	2-mm sieve mortar
a.	Air dry soil for at least 24 h.	•	pan for soil
b.	Grind soil with mortar and pestle or mechanical grinder.		
c.	Pass ground sample through 2-mm sieve to remove gravel.		
d.	Let the ground sample air dry for an additional 24 h (recommended).		

A Pretest soil sample can be used to characterize the organic matter content of the soil, calculate the sample dry mass, and perform any other pretreatment steps that may be needed, such as soluble salts and plasters removal or iron oxides removal (Section 2.3.1).

If no pretreatment is required (i.e., no organic matter or other matter needs to be removed), proceed to step 6 to determine the water content of the sample before running a PARIO measurement.

The PARIO measurement soil sample will be used for the actual PARIO measurement process and will go through the same treatment steps (Section 2.3.1) plus dispersion as additional step before the actual PARIO measurement begins (Section 2.3.1).

2.3.1 SAMPLE PRETREATMENT

The following steps describe the recommended procedure for pretreating the PARIO measurement sample prior to running the sedimentation experiment in the PARIO. Some of the pretreatment steps are critical where others are optional:

- Organic Matter Removal (optional depending on organic matter content)
- Soluble Salts and Plasters Removal (optional)
- Iron Oxide Removal (optional)
- Chemical Dispersion (required)
- Physical Dispersion (required)

To rely on the pretest results to calculate the net mass of soil used in PARIO analysis, it is important to treat the two sets of samples (pretest soil sample and PARIO measurement soil sample) in an identical manner.

Steps Equipment Needed

Prepare a Sample of Known Mass

precision scale (±0.01 g)

- a. Weigh out between 25 g and 50 g of soil from the 2-mm sieved, ground soil sample. If the soil sample has a high sand content, use more sample mass up to 50 g.
- b. Document the exact collected soil sample weight $(m_{air\ drv})$.

NOTE: Be precise with the dry-soil weight (within ±0.1 g) or the analysis will not be accurate.

NOTE: The usual samples are air dry and contain organic substances and other substances that can be removed during the pretreatment of the sample. In these cases, it is recommended to treat two samples in an identical way up to and including step 5. The mass loss experienced by one sample (pretreatment sample) is used to calculate the net mass of the PARIO sample.

Example calculation:

	Weight	% Total Mass
Initial weight of pretest soil sample (m_{wet})	50 g	100%
Dry weight of pretest soil sample (m_{dry})	47.5 g	95%
Weight of everything removed from pretest soil sample (water, organic matter, soluble salts and plasters, iron oxides)	2.5 g	5%

Net weight for PARIO measurement sample:

PARIO measurement sample weight	×	% dry mass of pretest soil sample	=	Total dry soil weight of PARIO measurement sample
30 g	×	0.95	=	28.5 g

NOTE: Ensure that the total dry soil weight of the PARIO measurement sample is greater than 25 g.

Steps (continued) **Equipment Needed**

Determine Organic Matter Content

Determine the percent of organic matter in the soil sample.

If the organic matter of the soil is unknown, use the most appropriate procedure for the circumstances (e.g., by following step 3 [see note below], using the brown coloring method, or by using an annealing method).

NOTE: The mass loss of the sample after organic matter is destroyed can be used to calculate percent of organic matter.

NOTE: Some procedures for determining organic matter are destructive and destroy the soil sample. If using one of these procedures, use a sample separate from the Pretest soil sample. In this situation, the separate soil sample will need to be discarded

b. If the organic matter content is >1.5% of the total dry mass, destroy the organic matter in the pretest soil sample as described in step 3.

Organic matter content can be estimated from knowledge of Organic Carbon content (OC) by multiplying OC with 1.72.

If the organic matter is <1.5%, proceed to step 4.

3. **Destroy Organic Matter**

MARNING: Read the respective safety data sheets for solids and chemicals carefully and follow them strictly. The following steps must be carried out with great care. Hydrogen peroxide (H₂O₂) can decompose violently with some organic substances that may be present in the soil. Never observe the reaction by looking into the glass beaker from above. Do not accelerate apparently slow reactions by heating or by adding any further H₂O₂.

These steps follow DIN ISO 11277 (2002) and Gee and Or (2002).

- Place the 25 to 50 g of soil sample from step 1 in a 1-L glass beaker.
- Document the exact sample mass for subsequent evaluations. h.
- Add 30 mL of distilled water to the beaker and stir with a glass stir rod until the soil and water are well mixed.
- d. Add 30 mL of 30% H₂O₂ and stir with a glass stir rod until well mixed.
- Cover the open beaker and let the sample soak overnight.
- f. Remove the cover.
- Put the glass beaker on a heating plate under a fume hood. g.
- h. Heat the solution at 40 °C, stirring the solution regularly. The solution will begin to foam.

1-L glass beaker distilled water glass stir rod 30% hvdrogen peroxide (H₂O₂) heating plate fume hood centrifuge

Steps (continued) Equipment Needed

 Watch the solution carefully because the foaming can occur suddenly and intensely.

- j. Add distilled water if the foaming gets too high.
 Take care that the sample does not burn in.
- Continue heating until the foaming due to decomposition of the organic matter has stopped, and existing bubbles have dissipated.
- l. This can take up to 4 h. Remove the beaker from the heating plate and let the sample cool down.

If the soil has a high organic matter content (>5% organic matter), the process of removing organic matter may need to be repeated 2 or 3 times.

If the soil does not have a high organic matter content, proceed to step m.

NOTE: Before adding more H_2O_2 , add distilled water to the soil sample, then remove residue by using a centrifuge, decanting, or by filtrating.

m. When all organic matter is destroyed, add distilled water and remove residue by using a centrifuge, decanting, or by filtrating.



MPORTANT: Make sure that the oxidation process of the sample is completed and the remaining H₂O₂ is expelled from the suspension before the PARIO measurement starts. If this is not the case, incorrect or even increasing pressures in the suspension due to the formation of gas during the measurement process may be observed. This will give inaccurate results.

4. Remove Soluable Salts and Plasters

In some cases it may be necessary to remove soluble salts and plasters. If the soil has saturated extract electric conductivity (EC) greater than 0.4 dS/m follow the steps in Appendix A to remove the soluble salts and plasters. If soluble salts and plasters are not removed, proceed to step 5.

5. Remove Iron Oxide

In some cases it may be necessary to remove iron oxides. If iron oxide coatings are present in the soil sample, follow the steps in Appendix A to remove iron oxides. If iron oxides are not removed, proceed to step 6.

Steps (continued) Equipment Needed

Step 6 should only be performed on the pretest soil sample.

6. Determine Dry Soil Weight (Pretest sample only)

The dry soil weight needs to be measured to account for the residual water content and the destruction of any organics, solubles salts and plasters, and iron oxides. This weight will be used to determine the true soil weight of the PARIO measurement sample.

- a. Record the sample weight plus the drying pan weight if it has not been recorded in previous steps.
- b. Transfer the pretest sample into a drying pan with known weight.
- c. Place the pretest soil sample and drying pan into a drying oven at 105 °C for 24 h.
- d. Weigh the dry sample plus the drying pan.
- Subtract the drying pan weight from the total pretest soil sample dry mass to determine the exact soil mass (m_{dry}).

NOTE: Be precise with the dry weight of the Pretest sample (within ± 0.1 g) or the analysis will not be accurate.

Example:

Pretest Sample Weight + Drying Pan Weight - Pan Weight =
$$M_{dry}$$

 $54.6 g$ - $7.8 g$ = $46.8 g$

precision scale (±0.01 g)

- drying pan, weighed
- drying oven

2.3.2 MEASUREMENT SAMPLE DISPERSION

The following steps describe the required dispersion procedure for the PARIO measurement sample prior to running the sedimentation experiment in the PARIO. These pretreatment are critical and therefore required:

- Chemical Dispersion (required)
- Physical Dispersion (required)

Sample Dispersion

There are two options for chemical dispersion of soil and two options for physical dispersion of soil (Section 3.4). Choose a method based on the available lab equipment and preferred standards

Step	os			Equipment Needed
СНЕ	EMIC	AL D	ISPERSION	
	Per	form	chemical dispersion prior to physical dispersion.	
	\triangle		NING: Read the respective safety data sheets for solids and chemicals ully and follow them strictly	
	ОРТ	ION	1 (DIN 2002)	distilled water
	a.	Pre	oare a 40 g/L solution of Na ₄ P ₂ O ₇ dispersing agent.	tetra sodium
		i.	Add 40 g of $Na_4P_2O_7 \times 10~H_2O$ into a 1-L Erlenmeyer flask.	pyrophosphate decahydrate
		ii.	Add enough distilled water to the flask to fill it to the 1-L mark.	(Na ₄ P ₂ O ₇ × 10 H ₂ O)
		iii.	Place the flask on a stir plate and let it stir until the $Na_4P_2O_7\times 10~H_2O$ is dissolved.	glass stir rod sealable 1-L
		iv.	Fill up the lost volume with distilled water.	bottle
		V.	Transfer the solution into a sealable 1-L bottle or dispenser.	shaker table
			NOTE: The solution will lose its effectiveness after about 1 month.	
			NOTE: Dry mass of pure Na $_4P_2O_7$ is only 20.27 g/L in this solution.	
	b.	Disp	perse the PARIO measurement sample.	
		i.	Place the PARIO measurement sample into either	
			 a sealable 1-L bottle (for physical dispersion OPTION 1), or 	
			- a 600-mL beaker (for physical dispersion OPTION 2).	
		ii.	Add 200 mL of distilled water to the bottle/beaker.	
		iii.	Add 1 mL of dispersing agent per gram of soil sample to the bottle/beaker.	
	0P1	ION	2 (Gee and Or 2002)	distilled water
	a.	Pre	pare a 50 g solution of $Na_6O_{18}P_6$ dispersing agent.	sodium
		i.	Add 50 g of $Na_6O_{18}P_6$ into a 1-L Erlenmeyer flask.	hexameta- phosphate
		ii.	Add enough distilled water to the flask to fill to the 1-L mark.	(Na ₆ O ₁₈ P ₆) glass stir rod
		iii.	Place the flask on a stir plate and let it stir until the $Na_4P_2O_7$ is dissolved.	sealable 1-L
		iv.	Fill up the lost volume with distilled water.	30000
		V.	Transfer the solution into a sealable 1-L bottle or dispenser.	
			NOTE: The solution will lose its effectiveness after about 1 month.	
			NOTE: To be sure about the dry mass of pure $Na_9O_{18}P_9$, it is recommended to experimentally determine the salt error (Section 2.3.2).	

OPERATION

Step	s			Equipment Needed
	b.	Dis	perse the PARIO measurement sample.	
		i.	Place the PARIO measurement sample into either of the following containers:	
			• a sealable 1-L bottle (for physical dispersion Option 1)	
			• a 600-mL beaker (for physical dispersion OPTION 2).	
		ii.	Add 250 mL of distilled water to the bottle/beaker.	
		iii.	Add 100 mL of dispersing agent to the bottle/beaker.	
PHY	SIC	AL D	ISPERSION	
	Phy	sica	l dispersion should occur after chemical the dispersion steps.	
		OP	TION 1 (DIN 2002)	shaker table
	a.	Sea	al the 1-L shaker bottle.	
	b.	Pla	ce sample on a reciprocating shaker and run for 6 to 12 h.	
		ME	TER recommends running this step overnight.	
		OP ⁻	TION 2 (Gee and Or 2002)	mixer cup
	a.	Allo	ow the sample to sit in covered 600-mL beaker overnight.	electric mixer
	b.		nsfer the sample from 600-mL beaker to mixer cup for persing with a wash bottle filled with deionized water.	
	C.		ce mixer cup in electric mixer (milkshake or ASTM mixer h soil-specific paddles) and run for 5 min.	

The sample is now prepared for the PARIO to measure (Section 2.4). If the soil sample needs to be stored, leave it in the used, sealable bottle.

NOTE: The effectivness of the dispersion will diminish over time. The sooner the sample is measured after these steps, the better the results.

2.4 MEASUREMENT

The PARIO measures the PSD of soil samples in suspension in a PARIO glass sedimentation cylinder (Figure 22).

In order for PARIO measurements to be accurate, pretreatment of the soil samples is required. Pretreatment involves the following:

- destruction of aggregates
- removal of binding agents
- dispersion of primary particles.

PARIO

Soils typically contain organic matter and, in some cases, iron oxides that cause the soil particles stick together. A proper pretreatment is required for any particle-size analysis (PSA) measurement.

METER recommends a sequence of steps for sample preparation that are presented in Section 2.3. The workflow described in this manual refers primarily to the German standard DIN ISO 11277 (DIN 2002) and Methods of Soil Analysis Part 4—Physical Methods (Gee and Or 2002), which are based on Stokes's law. The computation equations are only valid for a laminar settling of the particles as this is assumed for Stokes's law. There are other common and valid standards that can also be used (e.g., ASTM D7928) as well as chemical and physical pretreatment methods. The steps and sequence will vary depending on the standard.

After the soil sample preparation, the PARIO measurement process is divided into four parts:

- 1. Initialization (Section 2.4.2)
- 2. Execution of the measurement (Section 2.4.3)
- 3. Post processing (Section 2.4.4)
- 4. Evaluation and export (Section 2.4.5)

The samples, device, and suspension fluid must be equilibrated at room temperature. Room temperature should be constant within ±1.5 °C during the entire measurement. If possible, use a temperature-controlled room or place PARIO cylinders in a temperature-controlled water bath.

NOTE: Before starting the PARIO measurement, deactivate the computer standby mode and automatic updates to avoid interrupting tests.

2.4.1 PARIO CLASSIC VS. PARIO PLUS

PARIO measurements can be performed in two modes.

Classic mode Where the measurement process is fully automated after start of

the measurement and the measurement ends when the prescribed

measurement time is reached.

PARIO Plus mode Where the operator samples at some time a subsample of

the suspension by draining it through an outlet valve in the sedimentation cylinder. This sampling should be done after about 2.5 h (see STEP 4 in Section 2.4.3) and the measurement is finished.

In order to get maximum reliability and accuracy of results, METER recommends using the PARIO Plus mode because of the following advantages:

- The accuracy of the identified particle size distribution curve is higher, specifically for the clay fraction.
- The measurement time is considerably shorter.

• Errors in the particle size distribution (PSD) analysis that arise from uncertain or inaccurate external measured values (such as dry sample weight, sand fractions from sieve analysis), or disturbances (e.g., temperature fluctuations) are less problematic.

2.4.2 INITIALIZATION

Each PARIO device is stored in a cylinder filled with distilled water before and after the measurement. In this manual this cylinder is referred to as the standby cylinder. The cylinder with the soil sample in suspension to be tested is called the sedimentation cylinder.



IMPORTANT: All fluids, samples, and devices must be fully equilibrated at room temperature. Room temperature should be constant within ±1.5 °C during the entire measurement. If possible, use a temperaturecontrolled room or place PARIO cylinders in a temperature-controlled water bath..

STEP 1. Prepare workbench, standby cylinders, sedimentation cylinders, beakers, and PC Provide enough space on a workbench to place the desired number of standby cylinders, sedimentation cylinders, and beakers (PARIO Plus) next to each other on a flat surface. For a series of measurements with multiple PARIO devices, position the standby cylinders in a row behind the sedimentation cylinders, as viewed by the operator (Figure 2).

If the PARIO Plus mode is used, please do the following:

- Place the cylinder with the outlet opening in front and at a sufficient distance from the front edge of the worktable to be able to place a beaker in front of the sedimentation cylinders (Figure 2).
- Prepare a label for each sample beaker (180 mL to 200 mL, lightweight metal or glass) with the respective sample name and beaker weight. Make sure that the beaker is thoroughly cleaned and absolutely dry before weighing.

NOTE: Note the tare weight with an accuracy of 1/1000 g.

NOTE: If the surface material of a worktable is extremely hard (e.g., stone), it has proven advantageous to cover it with a thin rubber mat. This reduces the risk of damaging the PARIO glass cylinders.

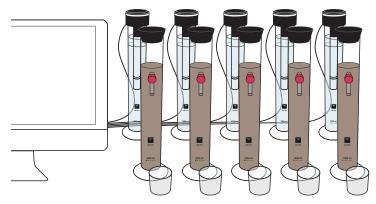


Figure 2 Schematic of workbench with standby cylinders, sedimentation cylinders, and beakers for PARIO Plus analysis

STEP 2. Prepare standby cylinders.

- 1. Fill the standby cylinders to 1 L with room-temperature distilled water.
- 2. Put the PARIO devices into the standby cylinders (Figure 3).

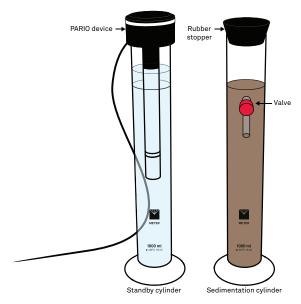


Figure 3 PARIO device and PARIO measurement sample equilibrating to room temperature

STEP 3. Prepare the sedimentation cylinders for the test.

- Transfer the dispersed soil sample into the sedimentation cylinder. Use a wash bottle with distilled water to ensure the whole sample is transferred.
- Fill the sedimentation cylinder with distilled water to bring the level of liquid to the 1,000-mL mark (Fill the sedimentation cylinder to the 1,000-mL mark), ensuring the meniscus touches the upper line of the mark.
 - Be sure that the distilled water is room temperature.
- 3. Place the rubber stopper on the sedimentation cylinder to avoid evaporation (Figure 3).

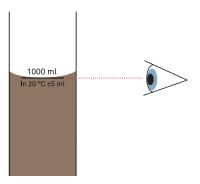


Figure 4 Fill the sedimentation cylinder to the 1,000-mL mark

STEP 4. Connect PARIO devices with the PC and start PARIO Control software.

- Connect the PARIO to the PC using the USB cable inserted into the USB port. If the number of USB ports on the PC is not sufficient for the number of PARIO devices, use a USB bank with an external power supply.
- 2. Start PARIO Control software.

The LED rings of all connected PARIO devices should flash white three times when the devices are detected, and then remain solid white to indicate the devices are ready for operation.

If this sequence does not happen, disconnect and reconnect the USB cable.



IMPORTANT: Before starting the PARIO measurement, deactivate the computer standby mode and automatic system updates to avoid interruption.



IMPORTANT: Make sure that the USB cables connecting each PARIO device to the computer are loosely aligned and free to move in the back of the workbench. They must be long and flexible enough to allow to transfer the PARIO device from the standby to the sedimentation cylinder without knocking over the other cylinders.

PARIO

When the software has started, the PARIO control screen opens up in a window. It is recommendable to set the respective window to full screen. The window has five sections (Figure 5).

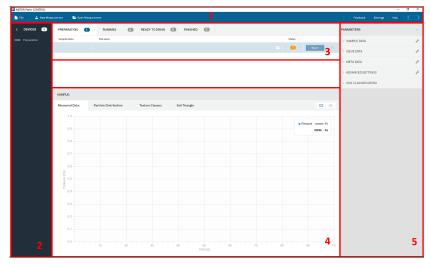


Figure 5 PARIO Control graphical user interface (GUI)

a. Top Panel

The top panel consist of eight menus that can be opened by clicking on the respective names. The left three menus are used to create new, open existing, and close open files; the right menus are used to get help, give feedback, and to define the default settings, including the definition and management of the sieve data templates. With the rightmost icons the user can open and close the left and right-side tab.

b. Left Panel

The left panel contains a list of the connected PARIO devices and current status of each device.

c. Open Measurement Panel

The open measurement panel lists the tests that are opened by PARIO control. The three tabs contain tests that are either in preparation status, currently running, or finished. Note that the different tests are automatically switched from one tab to another when the status of the test changes. If the vertical space is too small to show all the project, a scroll bar is displayed on the right side of the panel. Each of the tests can be selected by clicking on its name. The corresponding line will be highlighted in blue and the corresponding test will be displayed in the Current test field located below it.

d. Current Test Panel

The current test panel shows the figures of the measured data and results for the selected test. The panel has four tabs.

Measured Data tab—shows the measured data in real time for ongoing tests, specifically the pressure and temperature measurements. The temperature graph can be activated by right-clicking on the chart. For finished tests, it shows the fitted pressure curve and the evaluation results, namely the cumulative particle distribution curve, the texture classes as bar charts, and the resulting position of the sample in the texture triangle.

Two tabs can be shown at the same time. To switch between the view of one or two graphs, click on the respective icon on the upper right of the panel.

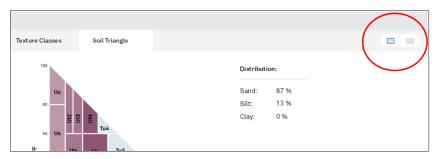


Figure 6 Icon to show one or two graphs

e. PARAMETERS Panel

This panel is used to configure the test parameters and to enter all required data for the evaluation of the measurements. It contains furthermore meta data for the respective test.

Expand the PARAMETERS side menu by clicking on the respective arrow. Five submenus show up. Each submenu can be folded and unfolded by clicking on the arrow in front of the tab name. Depending on the user's preference, when one section is opened, the other sections are automatically folded or remain open (Section 3.3.6).

NOTE: The vertical size of the list panel and the current test panel can be changed by the user. Point the mouse at the border of the two windows, hold down the left mouse button, and move the border line up or down.

STEP 5. Configure PARIO Tests

For each sample, do the following:

- 1. Click New Measurement on the PARIO Control main screen (Figure 7).
- 2. Enter Sample name parameter.
- 3. Change the automatically generated File name parameter, if desired.

Default values will be set in the windows in Figure 8, as predefined in the Settings menu (Section 3.3.6).

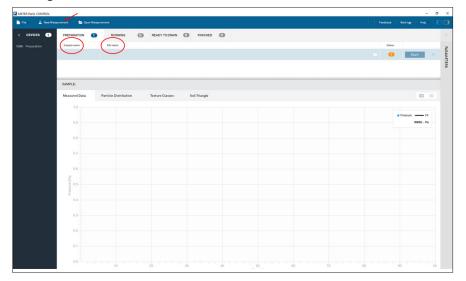


Figure 7 New Measurement screen

The following PARAMETERS are included in the SAMPLE DATA menu, Measurement Data section (Figure 8):

- Sample name
- File name
- PARIO Mode
- Ready to drain after (measurement duration or time until drainage)
- Counter for homogenization (homogenization time)
- Homogenization method (mixing mode)

OPERATION

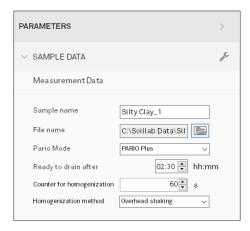


Figure 8 Parameters

- 4. Enter configuration parameters:
 - a. Sample name is already inserted
 - b. File name is not editable, because it has been set at the creation of the test.
 - c. Select the intended experiement PARIO mode.

This can either be PARIO Classic or PARIO Plus (default is PARIO Plus).

PARIO Classic mode:

The end of the PARIO run is defined by the selected Measurement duration. Evaluated data consist only of entered sieve data and recorded pressure data. Measurement time to identify particles down to clay size is 8 to 10 h. Estimate of the clay fraction is within $\pm 3\%$.

PARIO Plus mode:

The end of the PARIO run is defined by releasing a subsample of the suspension by the lateral valve. Typical time to identify particles down to clay size is about 2 to 3 h. Estimate of the clay fraction is within $\pm 0.5\%$.

5. Enter the Ready to drain after time length in hours.

PARIO Classic measurement duration in hours (default is 8:00 hrs).

The recommended measurement time in PARIO Classic mode is 8 hrs. In PARIO Plus mode, Measurement duration is not editable because the end of the measurement is defined by the manual drainage of part of the suspension after a specified time.

PARIO Plus ready to drain in hours—default setting is 2:30 h.

In PARIO Plus mode, the recommended measurement time is about 2.5 h. This field shows the time left until the sample can be drained. METER recommends a measurement time of at least 2 h. Upon release of the suspension (drain)

through the valve, the PARIO Plus measurement will automatically run for another 120 s, then stop. Draining of the suspension can be done at a later time, but METER does not recommend waiting longer than 12 h. If no release of suspension (drain) is recorded, the measurement will continue and stop after 24 h.

6. Enter the Counter for homogenization value—default is 60 s.

When starting the PARIO run, a countdown will take place for the duration of the entered time. During that time, the suspension in the sedimentation cylinder must be manually mixed. The homogenization time must be long enough to achieve a complete homogenization of the suspension at the begin of the sedimentation.

7. Select the Homogenization method.

The options are either Overhead shaking or Vertical stirring. Select the appropriate option.

IMPORTANT: Particles in the suspension often stick to the base. In that case vigorous shaking and/or stirring to mix up the slurry from the base of the cylinder is required before starting the countdown. Use visual inspection to be sure that all sediment is in suspension and no sediment sticks to the base before starting the countdown.

When these measurement parameters are entered, the basic configuration is finished, and the project is ready to start. This is indicated in the Preparation tab by a green checkmark in the Status column. The START button now becomes selectable and the LED ring will flash white (Figure 7).

NOTE: To run a PARIO Classic or a PARIO Plus measurement, no further parameters need to be set at this point. For the evaluation of the experiment, further parameters need to be entered. Known parameters (such as volume of suspension, particle density, dry mass of particles in suspension) can be inserted ahead of the PARIO test or alternatively after the PARIO run.

2.4.3 EXECUTION OF THE MEASUREMENT

STEP 1. Loosen sticky sediment from the sedimentation cylinder base.

If the mixing procedure chosen is Overhead shaking, close the sedimentation cylinder tightly with the rubber stopper so no suspension will be lost during the Overhead shaking. Perform premixing to ensure that no sediment sticks to the bottom of the cylinder.

STEP 2. Start the measurement.

In PARIO Control, click the START button for the measurement project. This initializes the timer for the homogenization countdown. During the countdown, a mixing animation window is shown in PARIO Control (Figure 9).

OPERATION

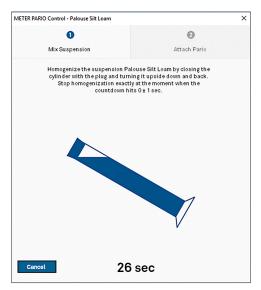


Figure 9 PARIO Control homogenization countdown with mixing instructions

- 8. Mix the suspension in the sedimentation cylinder thoroughly during the countdown using one of the following methods:
 - Continually turning the cylinder with the rubber stopper in place (Figure 10).
 - · Vigorous vertical stirring with a suitable stirrer (typically a plate with openings at the end of a bar).



IMPORTANT: Be absolutely sure to prepare a homogeneous suspension. Any error in the homogenization will propagate through the analysis and return wrong values.

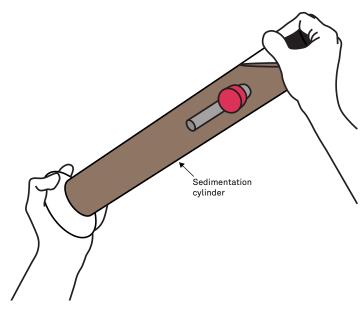


Figure 10 Mix soil sample

- 9. Place the sedimentation cylinder on the level surface when the countdown reaches 0 s (Figure 11).
- 10. Remove the rubber stopper from the sedimentation cylinder.

At this point the sedimentation starts.

The PARIO Control window will update to show instructions for placing the PARIO in the sedimentation cylinder (Figure 11).

NOTE: It is important that the cylinder is on a level surface when the countdown reaches 0 s $(\pm 1 \text{ s})$. The 0 s of the countdown initializes the start of the measurement. At this point the sedimentation has to start.

NOTE: Place the sedimentation cylinder with the valve facing front.

OPERATION

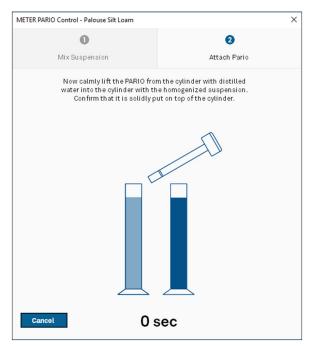


Figure 11 Completed PARIO Control countdown with instructions to insert PARIO into sedimentation cylindar

STEP 3. Insert PARIO device into sedimentation cylinder.

- 1. Remove the PARIO from the standby cylinder, bringing it momentarily to a horizontal position (Figure 12)
 - The short horizontal orientation is detected by the PARIO control software and identifies the PARIO device connected to the specific sample during the test.
- 2. Insert it into the sedimentation cylinder, carefully fitting the groove of the PARIO on the rim of the sample cylinder.
 - NOTE: Make sure the computer standby mode and automatic updates are deactivated to avoid interrupting the PARIO measurement.

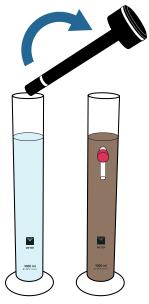


Figure 12 Insert PARIO device into sedimentation cylinder with soil suspension

3. Insert the PARIO into the sedimentation cylinder, carefully fitting the groove of the PARIO on the rim of the cylinder.

The amount of time from the end of the mixing until the insertion of the PARIO should not exceed 20 s because pressure data from time t=30 s will be used in the analysis.

If the device detection was successful, the following will happen.

- The measurement status line of the running measurement shown in PARIO Control is moved automatically from the Preparation tab to the In Progress tab (Figure 13).
- The PARIO LED ring changes to slow pulsing blue.

PARIO Control now records three parameters (time, pressure, and temperature) every 10 s and depects the recorded data graphically in real time (Figure 13).

If multiple PARIO devices are used, repeat STEP 1, STEP 2, and STEP 3for each sample. Switching windows in PARIO Control will not affect the running measurements.

4. Leave the PARIO devices undisturbed for the duration of the measurement time (Figure 13).

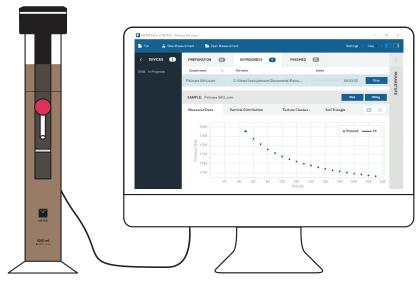


Figure 13 PARIO device and PARIO Control software during test

PARIO Classic Mode—In PARIO Classic mode, the end of the test is reached when either one of the following conditions occur:

- · The entered measuring time has expired, or
- The measurement is manually stopped by clicking the STOP button in PARIO Control.

PARIO Plus Mode—In PARIO Plus mode, the test ends 120 s after the suspension has been drained through the outlet valve of the sedimentation cylinder.

When the measurement in either mode is stopped, the following changes take place:

- The measurement status line of the running measurement shown in PARIO Control is automatically transferred from the Running tab to the Finished tab (Figure 14).
- PARIO LED will change to permanent blue when the measurement is complete. If using the PARIO Classic mode, skip STEP 4 and proceed with Section 2.4.4.

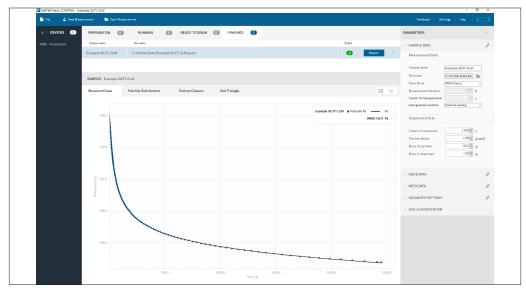


Figure 14 PARIO Control after PARIO Classic test

For short-term storage (e.g., the next measurement will be within a couple days), remove the PARIO from the suspension and place it again in the standby cylinder with distilled water (Figure 15).

If necessary, rinse the shaft of the PARIO with a soft jet of water and clean the shaft with a soft cloth. For long-term storage, see Section 4.2.

NOTE: Never touch the pressure sensor, which is shielded by the sensor cover. Spray only a soft stream of water in the area of the sensor protector to avoid damaging the pressure sensor.

STEP 4. Sample some of the suspension and determine its dry weight (PARIO Plus mode)

Between 2 to 3 h after the start of sedimentation, hold a beaker (document weight of beaker prior to this step) under the outlet opening, open the valve, and collect the draining suspension into the beaker. Draining should usually occur within 10 s after opening the valve. When draining is complete, close the valve again and place the beaker with the drained suspension in the drying oven. Dry at 105 °C until constant weight is reached (usually overnight).

PARIO Control automatically detects the draining because of the steep pressure drop and runs for another 120 s to safely record the dropped pressure, before it automatically stops the test.

After the drained suspension drying is complete, remove the beaker with the dry mass from the oven and place it in a desiccator to cool for a few minutes. Then place it on a balance and determine the weight with an accuracy of 1/1000 g. Calculate and document the dry sample weight.



IMPORTANT: Please be extremely cautious and precise when weighing this sample. METER recommends using a scale with a resolution of 0.0001 g and accuracy of 0.001 g, as used in pipette analysis. Use three positions after the decimal point. Ensure that the dry tare weight is truly reflecting the weight of the clean and dry beaker. Also ensure the weight of the beaker with the sample is completely dried. It is advantageous to have lightweight beakers, because then the difference of the two gross weights is less uncertain. Errors in weighing will directly lead to errors in the determined clay fraction of the soils..

2.4.4 POST PROCESSING

STEP 1. Return the PARIO device(s) to the standby cylinder(s)

After completion of the measurement, lift the PARIO device out of the suspension in the sedimentation cylinder and place it back in the standby cylinder with distilled water (Figure 15). If necessary, rinse the shaft of the PARIO with a gentle jet of water and clean the shaft with a soft cloth before inserting it into the standy cylinder with distilled water.



IMPORTANT: Never touch the pressure sensor that is shielded by the sensor cover at the end of the defice shaft. Spray only a soft stream of water in the area of the sensor cover to avoid damaging the pressure sensor.

NOTE: Since the PARIO device transfers a small amount of the suspension from the sedminentation cylinder to the standby cylinder, the distilled water may change over time and algae may form. In this case, METER recommends replacing the distilled water from time to time.

The PARIO devices in standby position are instantly ready for the next measurement. For short-term storage (e.g., when the next measurement takes place immediately afterwards or within a few days), the device can be kept permanently in the standby position. For information on long-term storage, see Section 4.2.

The suspension in the sedimentation cylinder (both PARIO Classic and PARIO Plus modes) can now be transferred to the sieves to determine the sand fractions using wet sieving.

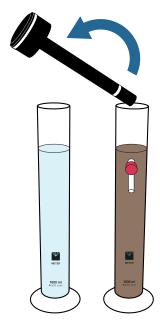


Figure 15 Replace PARIO in distilled water after test

STEP 2. Perform wet sieving.

Once the PARIO measurement is complete, the PARIO measurement sample needs to go through one final post processing step to determine the sand content. This is typically achieved by wet sieving the PARIO measurement sample.

Wet sieving of the soil sample has to be done to determine the sand fractions (2,000 to 63 or 50 µm) for the sample (Table 2). This can be done either with a homogenous parallel sample before or during the PARIO measurement or right after the measurement using the same sample measured with the PARIO.

NOTE: Sieving can be done with a separate sample of the same material. METER recommends doing the wet sieving right after the PARIO measurement using the same sample. This ensures that the results of the wet sieving (sand; Table 2) are consistent with the PARIO results (silt) and the clay fraction is correctly identified.

Table 2 Recommended mesh openings

Germany classification (µm)	USA classification(µm)
63	53
112	106
200	250
630	500
2,000	1,000

OPERATION

METER recommends doing the wet sieving right after the PARIO measurement using the same sample. This guarantees conformity of results from wet sieving (sand) to the PARIO results (silt) and, therefore, the correct identification of the clay fraction.

METER also recommends doing the sieve analysis with a three-dimensional working screening machine, combining throw pulse and angular momentum. This guarantees an optimal movement of the screenings.

The sieves are mounted downward with the largest mesh opening on top. Depending on the screening machine, the duration and type of movement have to be defined. Please refer to the appropriate screening machine manual.

Use the following steps to sieve the PARIO measurement sample (DIN 2002; DIN 2011).

- Transfer the contents of the PARIO sedimentation cylinder into the uppermost mesh of the screening tower.
- 2. Use a spray bottle to transfer all particles of the soil sample into the sieve.
- 3. Continue the screening until the water coming out of the drain hose is clear.
- 4. Place the residues of the sieves in labelled glass beakers.
- 5. Put the glass beakers in the oven for 24 h at 105 °C.
- 6. Calculate and document the dry soil weights to evaluate the wet sieving.

2.4.5 DATA EVALUATION AND EXPORT

Data evaluation is based on the integral suspension pressure (ISP) method (Section 3.4) or the extended integral suspension pressure method (ISP+) and PARIO Control software. All generated data can be exported into one Microsoft Excel® spreadsheet file, including visual illustration.

STEP 1. Enter Sample Data

These data evaluation steps can be performed in PARIO Control at any time during or after the PARIO measurement. In the Suspension Data subsection of the Menu SAMPLE DATA, enter the following data (Figure 16):

 In the Suspension Data subsection, enter the Volume of suspension value (default is 1 L) (Figure 16).

PARIO

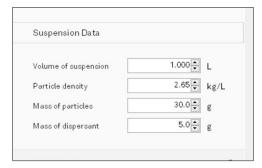


Figure 16 Example of evaluating data parameters

- 2. Enter Particle density value (default is 2.65 g/cm³).
- 3. Enter Mass of particles values (default can be specified in Settings).

The total dry soil weight is the total oven dry sample mass (105 °C for 24 h) including sand, silt, and clay.

The mass of particles entered for evaluation can be calculated by

- determining the water content with a Pretest sample (step 6, Section 2.3.1),
 and
- calculating the oven dry weight of the PARIO measurement sample (step 1, Section 2.3.1).
- Be cautious with respect to entering the correct value. If air-dry material is
 used and organic matter is destroyed, the respective amount of water and
 organic matter in the sample must be subtracted from the soil mass that has
 been actually used at the beginning of the measurement.
- Enter Mass of dispersant value (the total dry mass of dispersant in the 1-L suspension).

The dispersion mass is determined by adding a certain volume of dispersants solution to the soil. For example, if the dispersant solution = 40 g/L and the added dispersant volume = 25 mL, then the total dispersant in suspension = $40 \text{ g/L} \times 0.025 \text{ L} = 1.0 \text{ g}$.

For more information about these parameters, refer to Section 3.3.6.



MPORTANT: The exact mass of dispersion salt in the suspension is crucial for an unbiased determination of the clay fraction in the PARIO Plus mode.

Dispersant salts are usually traded in a formulation with water. In Germany, for example, Natriumpyrophosphat is generally provided as tetra-Sodium-diphosphate-decahydrate, Na₄O₇P₂ × 10 H₂O. The molar weight of this formulation is 446 g/mol. The corresponding dispersant solution is typically 0.1 molar (recommended), i.e., 44.6 g/L. However, weight of the dispersant after drying will be only 22.6 g/L.

Since dispersion salts are hygroscopic, the salt may have attracted moisture from the laboratory environment. If the dry mass of salt in suspension is not known for sure, METER recommends using a separate measurement to determine the salt error, as is common in the pipette analysis. To do this, put an aliquot (e.g., 50 mL) of the dispersion solution into a glass beaker and determine its mass after oven drying. For more information about these parameters and a calculation example, see Section 2.3.

Enter a value Dry mass in effluent (PARIO Plus mode only) 5.

The dry mass of the collected sample after oven drying (105 °C for 24 h) is the total mass minus the tare mass of the beaker used. The dry mass consists of dispersion salt and fine mineral particles. The proportion of dispersion salt is automatically calculated by PARIO Control and subtracted from the total dry mass to determine the dry mass of the fine particles in the drained suspension. Make sure to insert the accurate dry mass with three decimal places (Section 2.3.1).

STEP 2. Enter sieve data

In the SIEVE DATA Menu, enter the values for the sand fractions in percent of total mass into the table (Figure 17), which have been obtained from the sieve analysis. You can edit any entry in the table, add or remove rows in the table, and change the listed naming and values of the texture class size limits according your needs and to the available data.

The table with the user-defined classes and values can be saved as a template with a right mouse click. A saved template can be recalled anytime by left clicking on the tool symbol at the upper right.

NOTE: The last column weight refers to the weighting of the sieve points in the inverse analysis. If set to 0, it can be used to just visualize data points of the sieving but not considering it in the analysis. The last column is only visible if selected in Settings.



Figure 17 Enter sieve data

STEP 3. Select soil texture classification scheme

In the SOIL CLASSIFICATION menu, the default will be as specified in Settings, but can be changed for individual measurement runs. Select either US Soil Taxonomy or German Classification (KA 5, AG Boden 2005) (Figure 18). The texture classes, as well as the soil triangle will be adapted to the selected classification.

The texture classes as well as the soil triangle will be adapted to the selected classification setting.

NOTE: Shifting the size of silt/sand from 63 mm (German) to 50 mm (USDA) will change the identified sand and silt fractions.

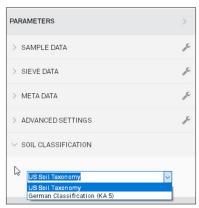


Figure 18 Soil Classification options

STEP 4. Perform data evaluation

The data that are evaluated consist of entered sieve data, recorded pressure and temperature data, and in mode PARIO Plus of dry total mass in the drained subsample. Minimum measurement time to identify particles down to clay size is 2 h for PARIO Plus and about 8 h for PARIO Classic.

 To evaluate the measurements, click on the Fitting button in the upper right of the current test panel. Alternatively, use the key combination Ctrl-F (Figure 19).

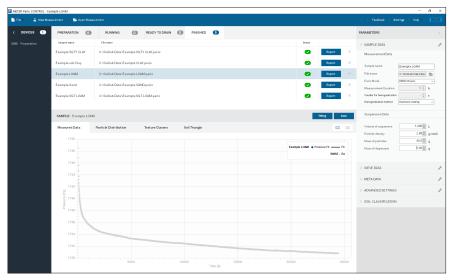


Figure 19 Fitting button above completed test

PARIO Control executes the fitting of the measured data. The fitting process can take several seconds and cannot be interrupted. The mouse icon will become an hourglass (or other icon) that indicates the computer is busy.

As documented in Durner, Iden, and von Unold (2017), the sedimentation process is simulated numerically, and the parameters that determine the cumulative PSD function are determined by nonlinear regression to achieve a best possible accordance between model and measurements (time series of pressure data by PARIO and independently measured sand fractions).

When the fitting is finished, the Fitting button will disappear, and the results of the PSD analysis are visible in the different tabs of the current test panel.

Notes:

- The result of the fitting depends on having the right settings, most importantly (1) the correct dry mass of mineral particles and (2) the correct sand fractions that stems from the independent sieving of the material.
- In the PARIO classic mode, any error in the sand fractions will propagate
 as complementary error to the clay fraction. PARIO identifies silt fractions,
 because sand particles settle so quickly that their behavior is not described
 by Stokes's law, and clay particles settle so slowly that most are still in
 suspension after 8 h.

PARIO

- Fitting is possible without entering sand fractions. However, in order to get accurate clay, sand, and silt calculations, the sand fraction data needs to be entered before fitting.
- If the initial pressure data do not show a decrease (due to the formation and disappearance of foam or other unexpected processes), PARIO automatically disregards these initial data. PARIO Control will shade the respective data gray.
- Data before time t=30 s are, by default, always disregarded.

As a result of the fitting, the parameters of the PSD are identified and interpolated by a Hermitian spline. The corresponding function is shown by PARIO Control and can be exported into an Excel spreadsheet file. By default, the depicted range of particle sizes extends from 2 µm to 2 mm. However, the PSD identification goes down to 0.1 µm and the respective data for the subclay range is given in the exported file.

2. Click Save to save the fitting in the location indicated in the File name column.

STEP 5. Export data and results

To export the measurement results, click Export from the Finished tab (Figure 20).



Figure 20 Export in the Finished tab

The exported Microsoft Excel file is subdivided in five tabs (Figure 21).

Settings & Parameters Shows all entered parameters.

•	•
Sieve Data	Gives the sieve data values.
Measured Data	Displays measured data points (runtime, measured and fitted pressure, temperature) and a chart showing runtime versus pressure versus temperature.
Particle Distribution	Shows the calculated cumulative PSD referring to particle diameter (in micrometers) versus cumulative relative mass.
Texture Classes	Gives final result of texture classes.
Soil Triangle	Displays the soil triangle with the soil type marked with a red point.
Diagnostics	Shows diagnostics of the model fit to the data, such as fitted number of data, weighed sum of square errors, mean error, root mean square error, mean average error, and modeling efficiency (R²). Also shows the values of the identified cumulative PSD at the supporting points and the depth of the sensor position in the suspension.

OPERATION

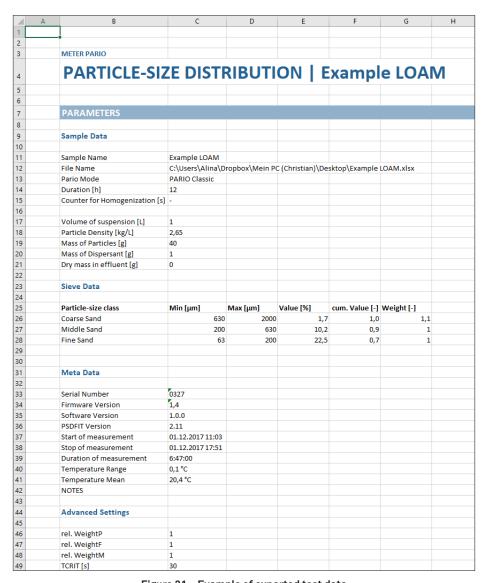


Figure 21 Example of exported test data

3. SYSTEM

This section describes the specifications, components, and theory of the PARIO.

3.1 SPECIFICATIONS

MEASUREMENT SPECIFICATIONS

Particle Size

Range 2-63 µm

Approximate Error in Mass Fraction Detection

PARIO Classic ±3.0% PARIO Plus ±0.5%

Pressure Measurement

Accuracy ±1.0 Pa

Resolution ±0.1 Pa

Typical Particle Mass

25-50 g per 1-L suspension

Typical Duration of Measurement

PARIO Classic 8.0 h
PARIO Plus 2.5 h

Measurement Interval

10 s

COMMUNICATION SPECIFICATIONS

Power Requirements

USB 5 V/100 mA

Computer Compatibility

Microsoft Windows 10

SYSTEM

PHYSICAL SPECIFICATIONS

Glass Cylinder	
Height	450.0 mm (17.7 in)
Diameter Inner Outer	59.0 mm (2.3 in) 67.5 mm (2.7 in)
Volume	1,000 cm³ (61.0 in³)
Material	Borosilicate glass 3.3
PARIO Device	
Height	293.0 mm (9.1 in)
Diameter	80.0 mm (3.2 in)
Material	Polyoxymethylene plastic (POM) and stainless steel
Volume of Susper	nsion

1,000 mL

±1.5 °C

Operating Temper	ature	
Minimum	15 °C	
Typical	20 °C	
Maximum	35 °C	
Maximum Tolerable Temperature Change During Measurement		

PARIO

Required External Measurements		
PARIO Classic	Content of organic matter (if OC is destroyed)	
	Sand fractions (from wet sieving)	
	Mass of dispersion salt in total suspension volume	
PARIO Plus	Mass of dry matter in discharged subsample	
	Sand fractions (from wet sieving)	
	Mass of dispersion salt in total suspension volume	
Cable Type		
USB 2.0; 500 mA	for receiving port	

COMPLIANCE

EM ISO/IEC 17050:2010 (CE Mark)

3.2 PARIO HARDWARE

The PARIO measuring system consists of the following components: PARIO device, two glass cylinders, rubber stopper, and glass beaker (PARIO Plus).

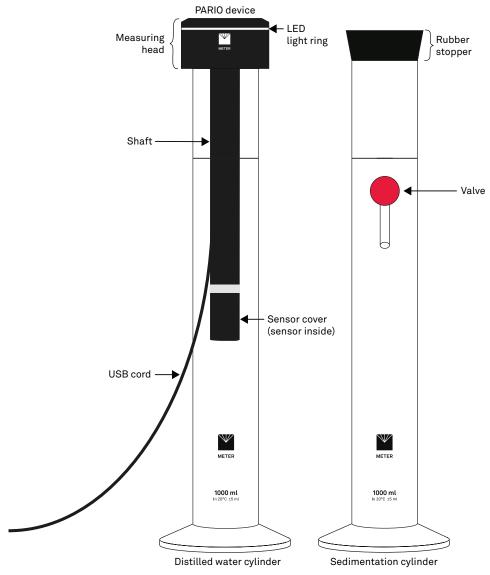


Figure 22 PARIO device

The PARIO device consists of a pressure sensor at the tip of a shaft, which is connected to a measuring head (Figure 22). A temperature sensor is located at the side of the pressure transducer. The pressure and temperature signals are processed in the measuring head on top of the sedimentation cylinder. The signals are transferred to a computer via a USB connection.

PARIO

The status of the instrument is indicated by the LED ring on the measuring head.

White flashing three times PARIO has been detected by the computer.

White permanent PARIO has been detected by PARIO Control and can be

configured for measurement.

White flashing PARIO is ready for measurement.

Blue slow pulsing Measurement is in progress.

Blue permanent Measurement is finished.

Red permanent Firmware is being updated.

Red flashing Cylinder or PARIO was bumped or moved.

This can also indicate a hardware error. Please contact

Customer Support.

No light PARIO is not connected to or recognized by a computer.

3.3 PARIO CONTROL SOFTWARE

The PARIO Control software is needed for configuration, measurement, and data manipulation. This section describes PARIO Control in detail.

The PARIO Control main screen is divided into five main parts (Figure 23):

- Main menu toolbar (Section 3.3.1)
- Device menu (Section 3.3.2)
- Test status tabs (Section 3.3.3)
- Parameters menu (Section 3.3.4)
- Test output windows (Section 3.3.5)

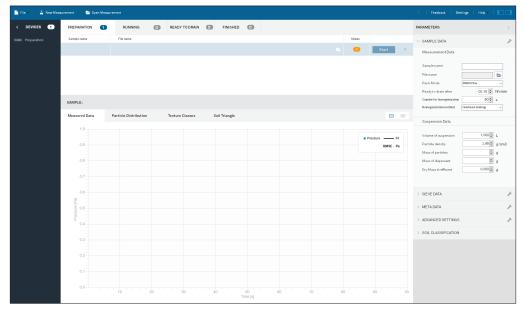


Figure 23 PARIO Control main screen

The main screen also provides access to the Settings window (Section 3.3.6).

3.3.1 MAIN MENU TOOLBAR

The Main menu toolbar contains main actions in the software (Figure 24).

File	Select menu for options to Save, Save All, Save as, Export selected, or Quit.
New Measurement	Click to begin a new PARIO measurement. It will populate in the Preparation tab to be configured.
Open Measurement	Click to open a finished measurement file (.pario). It will populate in the Finished tab after opening.
Settings	Click to open Settings window (Section 3.3.6).
Help	Click to open the PARIO user manual or information about the software. \\
Device menu collapse icon	Collapses the Device menu on the left.
Parameters menu collapse icon	Collapses the PARAMETERS menu on the right.



Figure 24 Main menu toolbar

3.3.2 DEVICE MENU

The device menu shows all connected PARIO device serial numbers and their current status (Figure 25). The Device menu can be collapsed or expanded by clicking on the arrow.

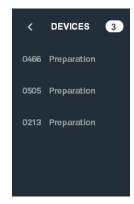


Figure 25 Device menu

3.3.3 TEST STATUS TABS

The test status tabs show all tests for all connected PARIO devices (Figure 26).

Preparation tab	New measurements
Progress tab	Running measurements
Ready to drain tab	Measurements in PARIO Plus mode that are ready to be drained
Finished tab	Completed measurements

The measurements are automatically moved from tab to tab as the software receives information from PARIO.

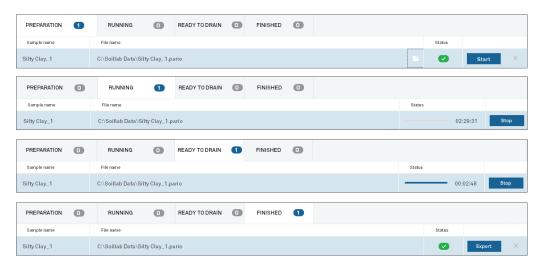


Figure 26 Test status tabs

Right click in the Test Status tab to close, begin, or open measurements (Figure 27).

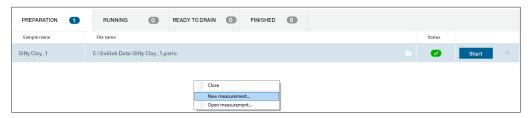


Figure 27 Right click menu in test status area

3.3.4 PARAMETERS MENU

The Parameters menu contains the following subsections (Figure 28):

- Sample Data
- Sieve Data
- Metadata
- Advanced Settings
- Soil Classification

The menu can be collapsed and expanded by clicking on it.

PARIO

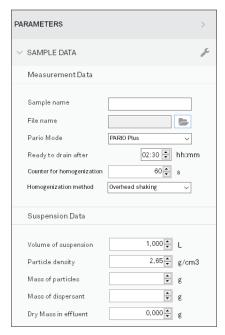


Figure 28 Parameters menu

SAMPLE DATA

The Sample Data subsection will be altered when setting up a new measurement (STEP 5 of Section 2.4.2). More in-depth explanations of the fields and the respective defaults are in Section 3.3.6. Commonly used fields and their definitions are:

Sample name Name can be anything the user chooses.

File name Field will be autopopulated.

PARIO mode Default is PARIO Plus.

Duration/Ready to drain after Default is 8 h in PARIO Classic mode and 2.5 h in PARIO

Plus mode.

Timer for homogenization Minimum recommendation is 60 s.

Homogenization method Default is overhead shaking

Volume of suspension Default is 1 L.

Particle density Default is 2.65 g/cm³.

Mass of particles The mass of particles is the total oven dry sample mass

(24 h at 105 °C) including sand, silt, and clay.

SYSTEM

Mass of dispersant The mass of dispersant in the 1-L suspension. Must be

either calculated or measured by a salt error blank sample.

Dry Mass in effluent Only in PARIO Plus mode.

The dry mass of matter in the drained suspension. The dry mass includes the particle mass and the precipitated

dispersant salt.

Clicking on the tool icon in the Sample Data header brings up three options:

Apply default settings Resets fields to defaults.

Save as default settings Saves field inputs as new defaults.

Copy to clipboard Copies data to the computer clipboard.

SIEVE DATA

The Sieve Data subsection is to enter sample sieve data (Section 2.4.4). The values should be entered in percents for the particle size classes. Clicking on the tool icon in the Sieve Data header brings up two options:

Example Sieve Data Autofills subsection with default example sieve data.

Copy to clipboard Copies data to the computer clipboard.

METADATA

The Metadata subsection displays the following information:

- Hardware serial number and firmware version.
- Software version
- PSDFIT version
- · Start of measurement
- Stop of measurement
- Duration of measurement
- Temperature range
- Temperature mean

Edits cannot be made to the information in this section. Clicking on the tool icon in the Metadata header gives the option to copy the information to the clipboard.

In the lower part of the METADATA submenu is a free text field, where user-specific meta data can be entered (i.e., operator name, laboratory, special occurrences, or other sample-specific text.

ADVANCED SETTINGS

The Advanced Settings subsection allows for changes to PARIO data evaluation and compensation parameters (Figure 29).



IMPORTANT: These settings should only be changed by very experienced users who know exactly what they are doing.

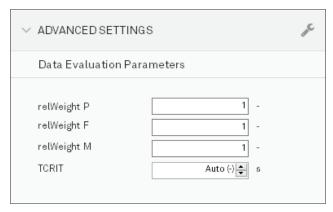


Figure 29 Advanced Settings subsection

In determining the PSD by inverse modeling, deviations between measured and simulated pressure data and between measured and simulated sand fraction data must be simultaneously minimized. The resulting object function that is to be minimized contains this data with different units, magnitude, and number. PARIO uses an internal weighing scheme. The user can modify this default weighing by increasing the importance of pressure data (relWeight P) or the importance of the sand fraction data from sieving (relWeight F). Increasing the weight of one data type may improve the fit to the respective data, but possibly worsen the fit to the other data type.

In the ISP+ method, additionally the mass of particles in the drained suspension is in the object function. Thus, in PARIO Plus mode also this weighting can be edited (relWeight M). Setting weights to zero completely disregards the data type in the inverse analysis. Increasing the weight of one data type may improve the fit to the respective data, but possibly worsen the fit to the other data type.

relWeight P

Default value is 1.

Relative weight of pressure data class in object function. Values can be arbitrarily increased or decreased, from 0 to 100. Entering 0 means that the pressure data are completely disregarded in the fitting process.

SYSTEM

relWeight F

Default value is 1.

Relative weight of sieve data class in object function. Values can be arbitrarily increased or decreased, from 0 to 100. Entering 0 means that the data class is completely disregarded in the fitting process.

relWeight M

Default value is 1.

Relative weight of the additional measurement of the released suspension. Value can be increased or decreased, from 0 to 100. Entering 0 means that the data class is completely disregarded in the fitting process.

TCRIT

Default is 30 s (AUTO).

Time of first data point to be used in the evaluation of the pressure time series.

Normally, PARIO records data beginning at about 20 s. This early pressure decrease is affected by particles of all sizes, including sand particles, which fall very quickly and possibly in a turbulent manner that cannot be described by Stokes's law. Thus, if the sample contains a considerable amount of sand, the very first pressure data should not be used in the PARIO analysis.

If early data show unphysical behavior (e.g., an increase in pressure), the AUTO mode will disregard the first data.

Compensation Parameters Compensation parameters are used to counteract distorting processes resulting from water flowing off the walls of the cylinder after mixing and temperature effects that may result from temperature changes during measurements, or temperature differences between the PARIO device and the suspension at the begin of the measurement. In PARIO Plus mode, scaling of observed pressure data compensates for mass balance conflicts, when externally entered sieve data and dry mass of drained suspension are not compatible with the recorded pressure drop. See Section 3.4 for details.

Deactivation of the compensation provides raw data of the pressure measurements which may lead to erroneous results of the inverse analysis.

It is NOT recommended to disable this field.

PARIO

Clicking on the tool icon in the Advanced Settings header brings up three options:

Apply default settings Resets fields to defaults.

Save as default settings Saves field inputs as new defaults.

Copy to clipboard Copies data to the computer clipboard.

SOIL CLASSIFICATION

The SOIL CLASSIFICATION subsection is used to select either the German soil classification KA 5 (defined in AG BOden, 2015) or USDA classification system.

3.3.5 TEST OUTPUT WINDOWS

PARIO Control provides four test output windows (Figure 30), which can be displayed in the sample window as a single tab or in two side-by-side displays. Double clicking in the data graphs opens the data table. The measurement data tabs can be printed, maximized, or exported by right-clicking.



Figure 30 Test output windows

MEASURED DATA

The Measured Data tab shows the recorded data of pressure versus time (Figure 31). Double-clicking in the data graphs opens the data table. The pressure axis can be toggled to show the temperature axis. The time axis can be switched between linear and logarithmic. Data can be shown either all or in a reduced amount. After fitting the pressure decrease, the fitted line is shown in superposition to the data.

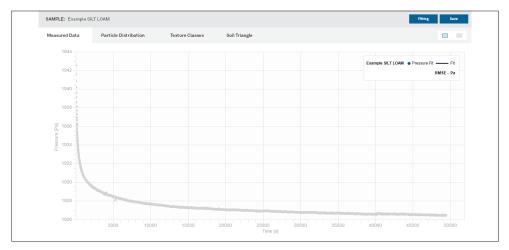


Figure 31 **Example Measured Data window**

PARTICLE DISTRIBUTION

The Particle Distribution tab shows the result of the analysis as cPSD, which is available after fitting the data (Figure 32), together with the external data for the cPSD.

NOTE: External data with weight zero (Section 2.3.5) are shown on the graph but not used in the fitting process.

This result is dependent on the chosen soil classification.



IMPORTANT: The total cPSD is correct only if the proper sieve fractions were inserted to the respective data window ahead of the fitting and if the inserted mass of dry mineral particles is correct.

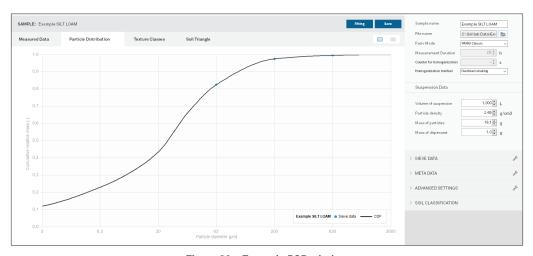


Figure 32 Example PSD window

TEXTURE CLASSES

The Texture Classes tab shows the mass fractions in two bar graphs. The left one shows the main classes clay, silt and sand. The right graph shows subcategories of clay, fine silt, middle silt, coarse silt, and the entered sand fractions (Figure 33). The last three bars show the added fractions for clay, silt, and sand.

NOTE: The fraction borders will depend on the selected classification scheme (Section 3.3.4). The size fractions will change accordingly if the classification scheme is changed.

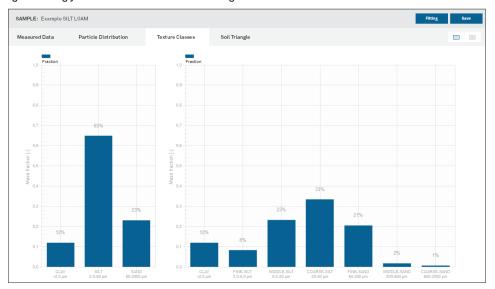


Figure 33 Example Texture Classes window

SOIL TRIANGLE

The Soil Triangle window shows the soil triangle and indicates the resulting soil type by a red dot (Figure 34). The user can switch between the German classification KA 5 (Boden, 2005) and the USDA classification system (Section 3.3.4).

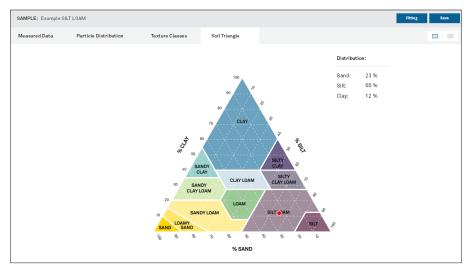


Figure 34 Example Soil Triangle window

3.3.6 SETTINGS WINDOW

The Settings window can be used to set various parameters. The updated parameters will be available for all subsequent individual measurements or can be selected to become defaults.

The settings window is categorized into three sections:

- General
- Default Parameters
- Sieve Data

GENERAL

The General settings are subdivided into Reports and Files (Figure 35).

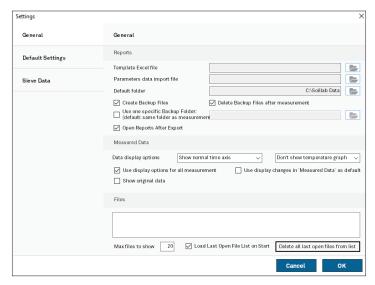


Figure 35 General settings

Template Excel file	Import a custom Microsoft Excel file (.x1sx). The PARIO data and evaluation will then be exported in the selected template.		
	NOTE: Expert use only.		
Parameters data import file	Import a user-generated Microsoft Excel file (.xlsx) including the sieve data.		
	NOTE: Expert use only.		
Default Folder	Select the saving location of the *.pario file.		
Open Reports After Export	Click the checkbox to automatically open reports in Microsoft Excel.		
Create Backup Files	Click the checkbox to generate a backup file. The backup file will be saved in the same folder as the measurement file.		
Load Last Open File List on Start	Click the checkbox to reopen all measurements that were last open when starting the software.		
Delete all last open files from list	Click to delete the listed files.		

DEFAULT PARAMETERS

The Default settings (Figure 36) allow the user to create new defaults for the Parameters menu (Section 3.3.4).

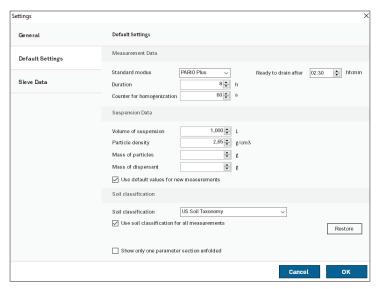


Figure 36 Default Parameters settings

Standard mode

Recommended mode: PARIO Plus

PARIO Classic mode: Measurement without release of part of the suspension through the sedimentation cylinder's outlet. Duration indicates end of the data recording and thus the end of the test. Recommended duration is 8 h. See Section 2.4.1 for basics and Section 3.4.5 for the theoretical background.

PARIO Plus mode: Measurement with additional release of part of the suspension. Duration of test normally is 2 to 3 h. See Section 3.4.6 for the theoretical background.

Ready to drain after

Affects PARIO Plus mode only.

Recommended: 2.5 h

When this test duration is reached, the respective test will be moved from the Running tab to the Ready to drain tab.

PARIO

Duration

PARIO Classic mode: Default 8 h.

The identified PSD depends toward the of small particles on the duration of the experiment. The uncertainty of the PSD is very small (down to approximately D = 2 μ m) for a duration of 8 h. At that time, particles of 2 μ m have reached a settling depth of about 10 cm and can be measured with the ISP method.

PARIO Plus mode: Default 24 h.

The identification of the PSD is controlled by the dry mass in the released suspension. If the release occurs after 2.5 h, the maximum particle size at the depth of the outlet is then about 3 μm . Since the released suspension also contains smaller diameters originating from the suspension above the outlet, the information content is very good to identify the clay fraction. The duration of the experiment in PARIO Plus mode is defined by the release of the suspension through the outlet of the cylinder. Thus, the Duration parameter indicates in this mode the maximum run time until this release can occur, which is set to 24 h.

Counter for homogenization

Recommended time is 60 s.

This parameter determines the mixing time of the suspension to produce a homogeneous suspension. When the measurement is started, a counter is initialized which counts down to time zero when data recording starts.

Volume of suspension

Recommended option is 1,000 mL.

A 1,000 mL suspension volume is recommended (DIN 2002; DIN 2011; ASTM 2007; ASTM 2017; ÖNORM 2002).

Mass of particles

Recommended dry mass is 25 g for fine textured soils and up to 50 g for sandy soils.

This value will mostly vary from test to test, since exact sample weights to 1/100th g are normally not prepared. Any default value can be set by the user.

See Section 2.3.1 for advice to calculate the dry sample weight.

Mass of dispersant

No default value is prescribed by METER. A default can be created by the user.

Typical values range from 0.5 to 1.5 g, depending on type, concentration and amount of dispersant solution added to the suspension.

Checkboxes

Use default parameters for new measurements	Click the checkbox to overwrite old defaults with new values.
Use soil classification for all measurements	Change soil classification for all measurement that are opened to the selected soil classification scheme.
Show only one parameter section unfolded	Opening a certain section in the PARAMETERS Menu will close all others.
Restore	Remove all user-entered values and reset the METER default values.

SIEVE DATA

The Sieve Data settings allow the user to edit, create, or import a sieve data template (Figure 37).

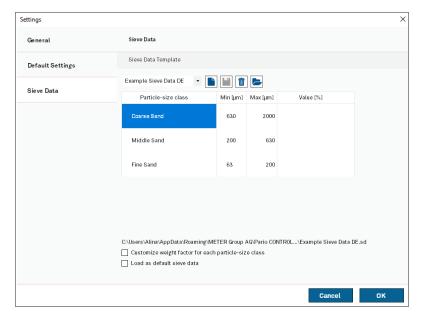


Figure 37 Sieve Data settings

The Example Sieve Data option sets the particle size classes in Table 4.

- Any sieve data table can be saved as a new sieve data template by clicking the save icon.
- Existing templates can be opened by clicking their name in the pop-up menu.
- Opened templates can be deleted by clicking the trash can icon.

Particle size classes can be added or deleted, and the minimum and maximum diameters of the chosen particle size classes can be freely defined.



IMPORTANT: Upper limits of the texture class with finer particles must be identical with the lower limit of the next larger texture class.

Table 4 Particle size classes

Sand Type	German Soil Mapping Guideline KA 5	USDA Classification System
Fine	63-200	50-250
Middle	200-630	250-500
Coarse	630-2,000	500-2,000

METER recommends using at least three sand subclasses (coarse, middle, and fine). If it is desired, other classes can be added by the user (e.g., splitting the fine sand fraction or lowering the limit of the finest sieved particles to 50 µm). Also, the determined values in percent can be entered here and used as default for other measurements if stored as a template.

The last column Weight refers to the weighting of the sieve points in the inverse analysis, NOT to weights in sieve analysis. Permissible values are 0 to 1000. If set to 0, sieve data are not included in the inverse analysis but can be used to just visualize data points of the respective particle classes.

The Weight column is only visible in the PARAMETERS menu (Section 2.4.4, STEP 2) if set here the respective tick mark Customize weight factor for each particle-size class (Section 3.3.6).

To apply a Sieve Data Template as default for new measurements, set the tick mark Load as default sieve data.

3.4 THEORY

This section describes the theory that supports the functionality of the PARIO.

PARIO is an automated system for PSA of soils. Soil is subdivided into fine soil (<2,000 µm) and gravel (>2,000 µm). Fine soil is further subdivided into sand, silt, and loam. The limits of the particle fractions are defined by equivalent diameters of the particles, which vary depending on national classification systems (Hartge and Horn 2014). PARIO Control uses the German soil mapping guideline KA 5 or the USDA classification system for the analysis (Table 5).

Table 5 Soil particle classification systems (Hartge and Horn 2014)

Particle Fraction	German Soil Mapping Guideline KA 5	USDA Classification System		
Sand	63-2,000	50-2,000		

Table 5 Soil particle classification systems (Hartge and Horn 2014)

Particle Fraction	German Soil Mapping Guideline KA 5	USDA Classification System		
Silt	2-63	2-50		
Clay	<2	<2		

The PSD of silt fraction ($2 \text{ to } 63/50 \ \mu\text{m}$) is obtained by the sedimentation method, which identifies silt fractions with hydrodynamic diameters between $63/50 \ \mu\text{m}$ (upper size limit) and $2 \ \mu\text{m}$ (lower size limit) (Section 3.4.1). The PSD for sand fractions is provided by external wet sieve analysis (Section 2.4.4). The clay fraction is calculated by PARIO Control for PARIO Classic mode, subtracting the externally measured sand and silt fractions from the total amount of material used in the analysis. In the PARIO Plus mode, the clay fraction is determined from the effluent measurement.

3.4.1 MEASUREMENT PRINCIPLE

PARIO uses the ISP method to derive the PSD from the pressure decrease at a measuring depth in a suspension. The theory of the method is published by Durner, Iden, and von Unold (2017) and is based on Stokes's law. Stokes's law indicates that particles of spherical shape settle in a suspension with a characteristic velocity, which is determined by the fluid viscosity, the density difference between the particle material and the suspension fluid, and the diameter of the particle. The computation equations are only valid for a laminar settling of the particles, as this is assumed for Stokes's law. The sedimentation methodology follows the requirements of DIN ISO 11277 (DIN 2002), ISO 13317 (ISO 2001), and ASTM D422-63 (ASTM 2007).

The preparation of the suspension involves destruction of organic matter and binding agents as well as dispersion of the material (Section 2.3). Users should refer to the respective operation protocols that are described in textbooks (e.g., Tan 2005), methods monographs (e.g., Gee and Or 2002), or normative documents (e.g., ISO 2001; ASTM 2007).

3.4.2 EFFECTS OF GAS BUBBLES

Gas bubbles may occur when the water in the suspension is not equilibrated with the ambient air temperature. Gas bubbles at the pressure transducer will affect its reading, and air bubbles that stick to liquid—solid interfaces may lead to bias in the weight of the water column above the measuring depth.

To avoid gas bubble formation, use slightly degassed water (e.g., by heating the deionized water prior to its use in preparing the suspension).

3.4.3 SAMPLE PRETREATMENT AND DISPERSION CONSIDERATIONS

For the measurements from the PARIO to be accurate, pretreatment of the soil samples is required. Pretreatment enhances the separation or dispersion of aggregates and is key for any PSA measurement. Soils typically contain organic matter and, in some cases, iron oxides that cause the soil particles to bind together.

The methods presented in this manual refer primarily to the German standard DIN ISO 11277 (DIN 2002) and Methods of Soil Analysis Part 4—Physical Methods (Gee and Or 2002). There are many other common and valid standards that can also be used (e.g., ASTM D7928) as well as chemical and physical pretreatment methods. For more details on the outlined steps, refer to the individual methods.

Sample preparation requires the same steps as the preparation for the hydrometer or the pipette method. Generally, it is the user's decision how to disperse soil samples, destroy binding materials, and remove organic matter. Typical procedures are described in the respective steps in Section 2.3.1 and may differ from discipline to discipline and country to country.

For more details on the outlined steps as well as the METER recommended sequence of the single steps, visualized in a flow chart, refer to Section 2.3.

3.4.3.1 ORGANIC MATTER REMOVAL

Removing organic matter from a soil sample is often one of the first pretreatment steps. The decision to remove the organic matter from the sample depends on the intended use of the analytical results of the PSA. A good rule of thumb is to remove organic matter if the organic matter is >1.5% of the total sample.

If the organic matter will be destroyed for the measurement sample, refer to Section 2.3.1.

3.4.3.2 SOLUBLE SALTS AND PLASTERS REMOVAL

A variety of soluble salts is commonly found in alkaline soils. Alkaline salts decrease the effectiveness of soil organic removal by decomposing hydrogen peroxide (H₂O₂), decreasing its effectiveness as a decomposing agent. Typically, if the extract electrical conductivity (EC) is >0.4 dS/m, METER recommends removing the salts. However, the decision to remove soluble salts and plaster is optional and up to the user (DIN 2002; Gee and Or 2002).

If soluble salts and plasters are to be removed from the measurement sample, refer to Appendix A.

3.4.3.3 IRON OXIDE REMOVAL

Iron oxide coatings often act as cementing and binding agents in soils. To properly disperse the silicate portion of soil, it may be necessary to remove these cementing agents. The decision to remove iron oxides is optional and up to the user (DIN 2002; Gee and Or 2002).

If iron oxides will be removed from the measurement sample, refer to Appendix A.

3.4.3.4 SAMPLE DISPERSION

There are many methods for dispersing soil aggregates. Chemical dispersion is achieved through the process of particle repulsion through the elevation of the particle electrokinetic potential (Soil Science Society of America, 1996). This process is typically achieved by saturating the exchange complex with sodium (Na). Physical dispersion involves the

separation of the individual soil particles by a mechanical or physical process. Sample dispersion is the last step done on a sample prior to PSA. Soil samples should be chemically and physically dispersed.

Please refer to step, Section 2.3.2 for recommended working steps (Option 1 [DIN 2002] and Option 2 [Gee and Or 2002]).

3.4.4 BASICS OF SEDIMENTATION METHODS

Sedimentation methods are the standard in soil science for determining particle size distributions (PSD) for particle diameters in the silt size fraction. The theory of the particle sedimentation method is based on Stokes's law, which indicates that particles of spherical shape settle in a suspension with a characteristic velocity. This velocity is determined by the fluid viscosity, the density difference between the particle material and the suspension fluid, and the diameter of the particle.

The theory is valid for a laminar settling of the particles. Therefore, the sedimentation method is suitable to identify the PSD for particles with an upper size limit of the hydrodynamic equivalent diameter of 63 μ m (German classification system) or 50 μ m (USDA system), resp., and 2 μ m as lower size limit. To get a full PSD from 2 μ m to the fine soil limit at 2,000 mm, it must be combined with results of an external sieve analysis.

The pipette method of Köhn (1928) and the hydrometer method (Casagrande, 1934) are sedimentation procedures that are established as standard methods of particle-size analysis (Gee and Bauder, 1986). A more recent method is the integral suspension pressure (ISP, Section 3.4.5) method, on which PARIO is based. Here, the suspension pressure is measured continuously at a depth of the sedimentation cylinder and the particle size distribution is determined by inverse simulation (Durner, Iden, and von Unold [2017]). Since ISP is based on the identical process, it is fully compatible with the traditional pipette and hydrometer methods, whereas alternative measurement methods, such as laser diffraction or photometric methods, may lead to incompatible results.

PARIO can be used to evaluate sedimentation experiments in two modes: The PARIO Classic mode, where ISP is applied without additional information about particle mass in the very fine silt fraction, and PARIO Plus mode, which uses additional information to improve the determination of the cumulative particle size distribution (ISP+ method, Section 3.4.6). The additional information in the released suspension regularizes the inverse problem and gives a more accurate and robust estimate of the cumulative particle size distribution at the fine particle diameter end. Together with a mass balance scaling (Section 3.4.7), use of the PARIO Plus mode improves the identification of the clay fraction greatly as compared to ISP.

3.4.5 THE INTEGRAL SUSPENSION METHOD (ISP)

The PARIO Classic uses the integral suspension (ISP) method. In a suspension, the settling of particles after an initial homogenization will lead to a pressure decrease in the suspension with time that is at any depth characteristic for the particle size distribution of the soil in suspension. In the ISP method, a mathematical model is used to calculate the pressure

decrease in a suspension at an observation depth as function of the PSD. The PSD of the analyzed sample is identified by fitting the simulated time series of pressure to the observed one by inverse modeling using global optimization. To identify from this a continuous curve of the PSD from the lower to the upper size limit, the PSD is expressed by a spline function and externally measured sieve data are included in the object function, given by Equation 1:

$$O_{\mathit{ISP}}\!\left(\theta\right)\!=\!\frac{1}{\sigma_{P}^{2}}\!\sum_{i=1}^{^{n_{P}}}\!\!\left[P_{i}\!-\!P\!\left(t_{i},\theta\right)\right]^{2}+\frac{1}{\sigma_{F}^{2}}\!\sum_{i=1}^{^{n_{F}}}\!\!\left[F_{i}\!-\!F\!\left(D_{i},\theta\right)\right]^{2} \qquad \qquad \text{Equation 1}$$

Here, $p(t_i,\theta)$ [Pa] and $F(D_i,\theta)$ [—] denote simulated suspension pressures and point values of F(D) in the sand range (according to the German classification $D \ge 63~\mu m$ (AG Boden, 2005); according to USDA classification $D \ge 50~\mu m$ (Gee and Or, 2002)), which depend on the parameter vector θ . The sand fractions F_i in Equation 2 must be determined independently by sieving, since Stokes's law requires a laminar settling of particles, which is fulfilled for particles in the silt and clay fraction but not for the sand fraction. The squared sums of deviations must be normalized with the expected variances σ_p^2 and σ_F^2 , which essentially reflect the assumed measurement accuracy of the respective data and fixed in PARIO. A user may overrule the default weights by changing the relative weights in the optimization, as listed in Section 3.3.4 (Figure 29). The PSD is determined by iteratively minimizing the objective function using global optimization. For details, the reader is referred to the open access publication of Durner, Iden, and von Unold (2017). The ISP method has been implemented in PARIO Classic. This implementation leads to a computerized system which yields a quasi-continuous particle-size distribution curve.

3.4.6 THE EXTENDED INTEGRAL SUSPENSION METHOD (ISP+)

PARIO Plus uses an improvement of the ISP method referred to as the extended integral suspension (ISP+) method which is achieved by the additional measurement of the total dry mass in a partial volume of the suspension. By using a sedimentation cylinder with a side outlet, a fraction of the entire suspension is sampled from a certain depth after a certain time and its total mass is measured after oven-drying. The inclusion of the particle mass into the objective function regularizes the inverse problem (Equation 2):

$$O_{\rm ISP}\!\left(\theta\right)^{\!\!\!\circ} = O_{\rm ISP}\!\left(\theta\right) + \frac{\left(m_{\rm sim} - m_{\rm obs}\right)^2}{\sigma_{\rm m}^2} \qquad \qquad {\rm Equation \, 2}$$

where m_{sim} is the simulated dry mass of particles and dispersant, contained in the drained suspension, and σ_m^2 is the variance of this mass. This improved method is called ISP⁺ (read as ISPplus). The gain in accuracy and potential reduction of measuring time by ISP⁺ as compared to the ISP is considerable.

ISP⁺ has been implemented into PARIO Control. The corresponding evaluation procedure PARIO Plus uses the following calculations:

1. The end of the PARIO run is defined by the time when a partial volume of the suspension fluid is released through the outlet of the sedimentation cylinder (typically 2 to 3 h).

- 2. This time of suspension release is automatically detected by PARIO from the corresponding big pressure drop.
- 3. The volume of suspension that has been released is automatically calculated from the magnitude of the pressure drop and the geometric data of the PARIO system.
- 4. The value for the total dry mass in the released suspension must be inserted by the user into the PARIO Control interface. It is then used to calculate the dry mass of particles, automatically subtracting the mass of dispersion salt.
- 5. The dry particle mass is included in the object function of the parameter estimation procedure, which estimates the full PSD based on the recorded pressure, plus the externally added information of sand fractions and dry mass in the released suspension.
- If a mass balance conflict is detected between the clay mass (indirectly calculated from particle mass in the released suspension), silt mass (calculated from pressure data) and sand mass (from sieving), PARIO Plus scales the pressure data to achieve a proper mass balance (Section 3.4.7).

3.4.7 MASS BALANCE CLOSURE IN THE PSD ESTIMATION

The pressure decrease in the observation depth is dependent on the total mass of silt particles in the suspension. The externally measured sand fractions and the measured total dry mass in the outlet, m_{obs} , constrain the possible pressure decrease. Any error in either of these variables will lead to an incompatibility between the observed and the simulated pressure decrease data. A perfect fit of the observed data will not be possible. Similarly, deviations from the assumptions with respect to the density of solids, errors in the total dry mineral particle mass (Section 2.3.1), or errors in the calculation or measurement of the dry mass of dispersion salt (Section 2.2.3) will make a perfect fit of the observed data impossible.

As a consequence, depending on the weighting of the three data types in the object function (sieve fractions, pressures, m_{obs}), the identified PSDs will be different, i.e., there is a pareto optimum. Practical experience with PARIO has furthermore shown that water running off the cylinder walls and temperature effects at the beginning of the measurement, when the PARIO device is not perfectly in equilibrium with the temperature of the suspension can cause distorted pressure data. Both effects diminish relatively quick, but they might vary from run to run. This suggests that to correct the observed pressure data p_i in Equation 1, add a scalable correction function (Equation 3):

$$p_i^* = p_i + p_0 \times t_i^\beta$$
 Equation 3

Experiments with pure water, where temperature effects and the effect of draining water from the cylinder walls had been specifically tested, led to the conclusion that a power function with exponent $\beta = -1.2$ characterizes the time scale of the two effects quite well. Thus, we resolve the pareto optimum problem by including the scaling coefficient p_0 in the parameter vector. Of course, this implies that errors in the sieve fractions, particularly the

fraction of fine sand, and errors in m_{obs} will lead to an erroneous correction of the observed pressure decrease. In addition, the correction function does not capture the systematic bias that would be caused by incorrect density of solids in the suspension.

Experienced users can change the weighing factors for the three data types in the object function, as described in Section 3.3.4.

3.4.8 TEMPERATURE EFFECTS

Temperature affects the sedimentation of particles, because viscosity and density of water are temperature dependent. Dynamic viscosity is approximated by the function $y = 0.0007 T 2 - 0.0531T + 1.764 (r^2 = 0.9996)$, where T is given in degrees Celsius. Table 6 lists some characteristic values of the viscosity at various temperatures.

Table 6 Characteristic values of water viscosity at various temperatures

Temperature (°C)	5	10	15	20	25
Dynamic viscosity of water (mPa·s) at 1 bar	1.518	1.306	1.137	1.001	0.894

PARIO Control records the temperature during a run, calculates the mean temperature, and uses this mean temperature for its calculations. Temperature changes during measurement run should thus be avoided. Ideal are measurements in a temperature-controlled room.

Temperature changes have also implications of the pressure recordings. Slow changes in room temperature are tolerable, because the effects of fluid expansion and device expansion upon warming (or shrinking upon cooling) are corrected by a compensation function (Section 3.4.9). Problematic are temperature differences between the PARIO device and the suspension temperature that might occur at the beginning of the measurement. They should be avoided (Section 3.4.5), because their influence on measurement results, particularly in PARIO Classic mode, might be severe.

NOTE: PARIO Plus mode makes measurement more robust with respect to initial temperature drifts.

3.4.9 COMPENSATION OF TEMPERATURE AND DRAINAGE EFFECTS

Liquid expansion

The temperature during a sedimentation analysis should be kept as constant as possible. However, moderate temperature drift is common in labs that are not temperature controlled. These changes are permissible withing a bandwidth of 3 °C and recorded by PARIO. Whereas the PARIO pressure sensor is temperature compensated, different thermal expansion of liquid and solid parts of the experimental setup will cause a change of the hydrostatic pressure, which is not due to particle sedimentation. To compensate for this, PARIO corrects the recorded raw data by adding $\Delta p = 1.3~{\rm Pa}/{\rm °C} \times \Delta T$ (i.e., a pressure head change of 0.13 mm/°C), where ΔT is the temperature difference with respect to the mean temperature of the measurement run.

SYSTEM

Temperature drift

Initial temperature differences between suspension and PARIO device will lead to an extension or shrinking of the PARIO device shaft and a small counteracting process in the liquid. The temperature differences between PARIO and the suspension typically disappear in an exponential function during the first 300 s. This process is compensated by adding an empirical compensation function $\Delta p=a\times\Delta T^3+b\times\Delta T^2+c\times\Delta$ with coefficients that have been determined by METER with pure water.

Drainage

As a result of the mixing, a certain mass of water is present as a water film on the walls of the cylinder at time zero. This water drains in the first seconds down and adds pressure to the free suspension, which is recorded by PARIO. Since this pressure increase is not due to the process of particle settling, it is compensated in PARIO by subtracting an empirical drainage power function $\Delta p = a \times t^{-b}$. The parameters have been empirically determined by METER. They are a = 35 Pa and a = 1 s^{-1} for overhead shaking, and a = 1 Pa and b = 0.5 s^{-5} for vertical stirring.

4. SERVICE

This section describes the calibration and maintenance of the PARIO. Troubleshooting solutions and customer service information are also provided.

4.1 CALIBRATION

Each PARIO device is temperature and pressure calibrated after manufacturing. METER recommends sending the device for calibration verification every 2 years. In case of conspicuous errors, PARIO Control will give a warning message to send the PARIO back for an inspection by METER. Contact Customer Support with questions.

4.2 MAINTENANCE AND STORAGE

To clean the exterior of the PARIO, wipe it with a damp cloth. Avoid leaving pools of water on the device as this will lead to salt precipitates. If fine soil and sand grains are deposited in the socket and settle into the base at the end of a measurement, clean the entire base under a gentle spray of water. The pressure transducer of PARIO is ultra-sensitive, so avoid contact with hard, sharp objects or a strong water jet.

For short-term storage, METER strongly recommends placing the PARIO device in a PARIO standby cylinder that contains 1 L of degassed distilled water, equilibrated to the room temperature at all times.

For long-term storage, METER recommends rinsing all parts of the device with distilled water to remove soil particles and prevent the formation of algae. Store the device in a dry state.



IMPORTANT: Never touch the pressure sensor, which is shielded at the tip of the PARIO shaft.

4.3 TROUBLESHOOTING

Table 6 lists common problems and their solutions. If the problem is not listed or these solutions do not solve the issue, contact Customer Support.

Table 6 Troubleshooting the PARIO

Problem	Possible Solutions
PARIO does not flash three times when connected or Computer does not recognize the device	Disconnect and reconnect the USB. Check the computer Device Manager to see if the PARIO USB driver is installed correctly. Reinstall the PARIO USB driver, if necessary. Restart the PARIO Control software. Check that the USB hub has sufficient power.

REFERENCES

Table 4 Troubleshooting the PARIO (continued)

Problem	Possible Solutions
Start of measurement was not recognized automatically when inserting PARIO into the cylinder	Close the countdown window. Place the device back into the standby cylinder. Unplug and replug the USB into the computer. Press Start and homogenize the sample again.
Computer goes into standby mode and interrupts measurement	Deactivate the standby mode in the computer settings and start measurement again.

4.4 CUSTOMER SUPPORT

NORTH AMERICA

Customer service representatives are available for questions, problems, or feedback Monday through Friday, 7:00 am to 5:00 pm Pacific time.

Email: support.environment@metergroup.com

sales.environment@metergroup.com

Phone: +1.509.332.5600
Fax: +1.509.332.5158
Website: metergroup.com

EUROPE

Customer service representatives are available for questions, problems, or feedback Monday through Friday, 8:00 to 17:00 Central European time.

Email: support.europe@metergroup.com

sales.europe@metergroup.com

Phone: +49 89 12 66 52 0 Fax: +49 89 12 66 52 20

Website: metergroup.de

If contacting METER by email, please include the following information:

Name Email address

Address Instrument serial number
Phone Description of the problem

NOTE: For products purchased through a distributor, please contact the distributor directly for assistance.

4.5 TERMS AND CONDITIONS

By using METER instruments and documentation, you agree to abide by the METER Group, Inc. Terms and Conditions. Please refer to metergroup.com/terms-conditions for details.

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Tan, Kim H. 2005. Soil Sampling, Preparation, and Analysis. Boca Raton: CRC Press.

APPENDIX A. NONTYPICAL SOIL PRETREATMENTS

In some cases, additional pretreatment of the soil prior to analysis with the PARIO may be required.

A.1 SOLUBLE SALTS AND PLASTERS REMOVAL

It is typically recommended to remove soluble salts and plasters if the saturated soil sample extract electrical conductivity (EC) is >0.4 dS/m. There are multiple methods available for measuring the EC of the sample. A saturated extract EC is one of the simple approaches to determining the sample EC (Rhoades 1996).

If the EC is >0.4 dS/m, proceed to step 1 below and remove soluble salts and plasters in the Pretest soil sample and PARIO measurement sample using either OPTION 1 (DIN 2002) or OPTION 2 (Gee and Or 2002).

If the EC is less than 0.4 dS/m, no removal is necessary.

Step		Equipment Needed	
1.	Det	termine if EC is > or < 0.4 dS/m.	device for measuring EC
2.	Rer	nove Soluble Salts and Plasters	
	Below are two different options for removing soluble salts and plasters from soil. The methods chosen may vary depending on the equipment available in the lab and the standards followed.		
	OP	TION 1 (DIN 2002)	centrifuge
	a.	Transfer the sample after organic matter removal (step 3, Section 2.3.1) to a 1.5-L bottle.	device for measuring EC
	b.	Add distilled water to the bottle until the ratio of soil to water is 1:4 or 1:6, according to the percentage of soluble components.	
	c.	Close the bottle and shake until everything is in suspension.	
	d.	Place on an overend shaker or alternative piece of equipment and shake the bottle for another hour.	
		NOTE: Use a vibration blade agitator if no overend shaker is available. Mixing agitators should not be used as they could crush the primary particles.	
	e.	Put the closed bottle on a centrifuge for 1 h until the supernatant is clear.	
	f.	Measure the EC of the clear supernatant.	
		If the EC is >0.4 dS/m, decant the supernatant liquid.	
	g.	Open the bottle, add 250 mL of distilled water and close the bottle.	

Step		Equipment Needed
h.	Put the closed bottle on an overend shaker or alternative equipment and shake for 1 h.	
i.	Remove the bottle from the shaker.	
j.	Put the bottle on a centrifuge and run until the supernatant is clear.	
k.	Repeat this procedure (step e through step j) until the EC of the liquid is <0.4 dS/m.	
l.	Finally decant the supernatant liquid.	
OPTION 2 (Gee and Or 2002)		distilled water
a.	Place filter paper (e.g., Whatman #4) in a Büchner funnel or standard funnel large enough to hold the sample and water.	filter paper funnel
b.	Transfer the soil sample after organic matter removal (step 3, Section 2.3.1) to the filter paper using a wash bottle with deionized water.	
C.	Fill the funnel with deionized water and let drain.	
	Do not let the water rise above the filter paper. Repeat 3 or 4 times.	
d.	Check the EC of the extracted water. Repeat step c until the EC is <0.4 dS/m.	

A.2 IRON OXIDE REMOVAL

Iron oxide coatings often act as cementing and binding agents in soils. To properly disperse the silicate portion of soil, it may be necessary to remove these cementing agents. The decision to remove iron oxides is optional and up to the user (DIN 2002; Gee and Or 2002).

Steps		Equipment Needed
Below are recommended steps for removing iron oxides from the soil sample.		sodium acetate solution
WARNING: Read the respective safety data sheets for solids and chemicals carefully and follow them strictly.		(NaOAc) acetic acid (AcOH)
a.	Use approximately 0.3 mol/L sodium acetate (NaOAc) solution, buffered with acetic acid (AcOH) to pH 3.8.	sodium dithionite
b.	Add 40 g/L $Na_2S_2O_4$ to the buffered solution.	(Na ₂ S ₂ O ₄) centrifuge
c.	Add the mixture to the sample until the ratio of soil to solution is 1:40.	

Steps		Equipment Needed
d.	Place the sample in 1.5-L/1-L shaker bottle.	
	Place 1.5-L/1-L shaker bottle on reciprocating shaker.	
e.	Shake the soil overnight.	
f.	The following day, shake the suspension on a centrifuge until the supernatant is clear.	
g.	Decant the clear liquid.	
h.	Collect waste separately.	
i.	Add distilled water, stir and allow the sample to settle again.	
j.	Discard the supernatant again.	
k.	Repeat these steps until the supernatant is clear.	

APPENDIX B. EXAMPLE MEASUREMENT RESULTS

B.1 EXAMPLE SILT LOAM

Figure A.1 through Figure A.4 show a typical measurement of silt loam sedimentation.

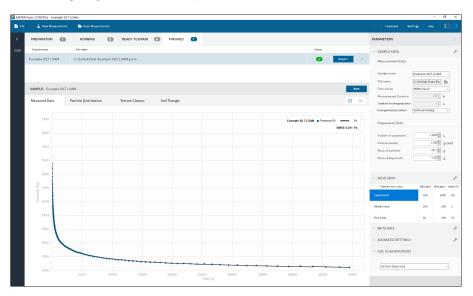


Figure A.1 Example silt loam measured data results

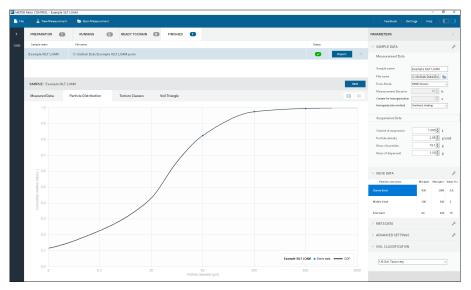


Figure A.2 Example silt loam particle distribution results

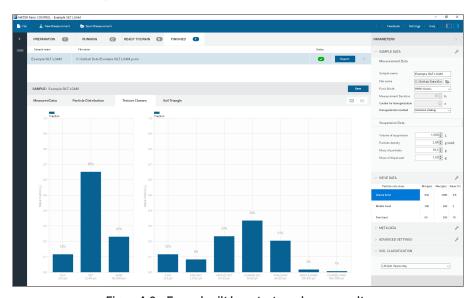


Figure A.3 Example silt loam texture classes results

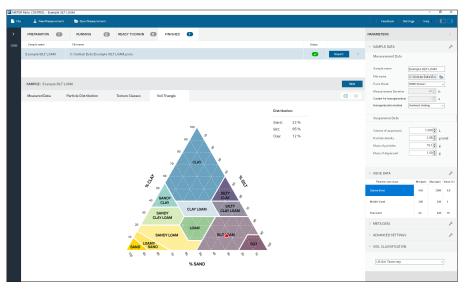


Figure A.4 Example silt loam soil triangle results

B.2 EXAMPLE LOAM

Figure A.5 through Figure A.8 show a typical measurement of silt loam sedimentation.

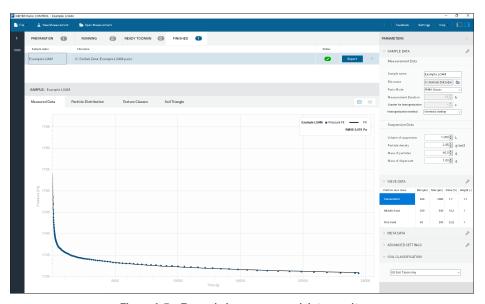


Figure A.5 Example loam measured data results

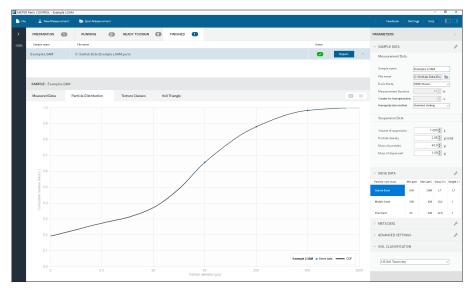


Figure A.6 Example loam particle distribution results

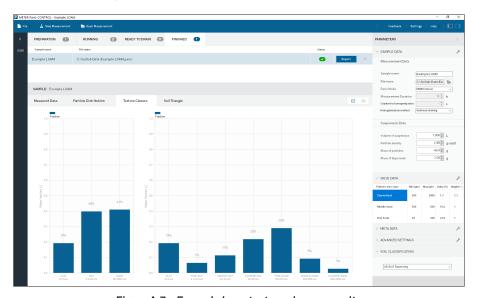


Figure A.7 Example loam texture classes results

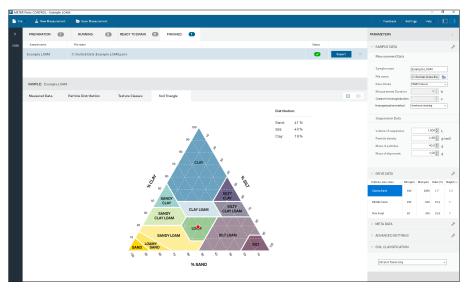


Figure A.8 Example loam soil triangle results

B.3 EXAMPLE SILTY CLAY

Figure A.9 through Figure A.12 how a typical measurement of silty clay sedimentation.

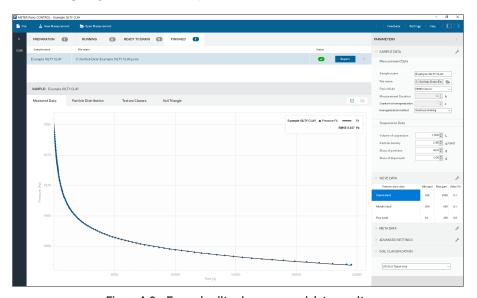


Figure A.9 Example silty clay measured data results

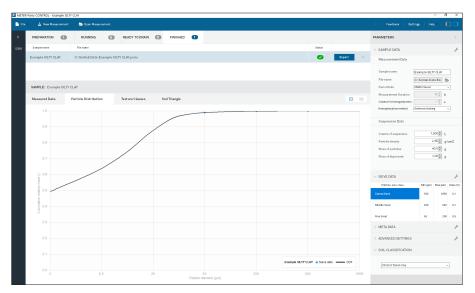


Figure A.10 Example silty clay particle distribution results

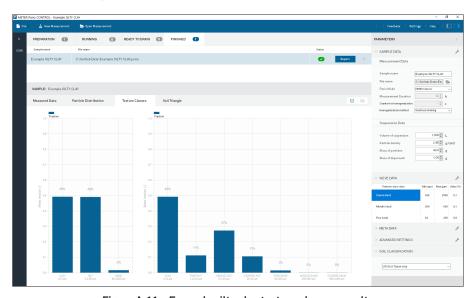


Figure A.11 Example silty clay texture classes results

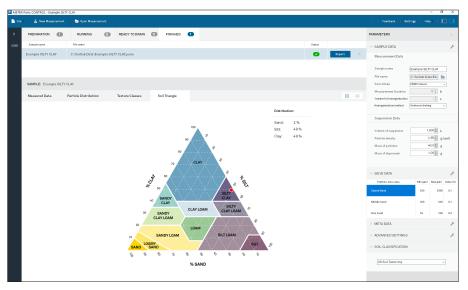


Figure A.12 Example silty clay soil triangle results

B.4 EXAMPLE SAND

Figure A.13 through Figure A.16 show a typical measurement of sand sedimentation.

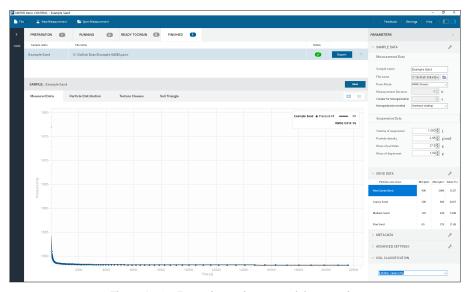


Figure A.13 Example sand measured data results

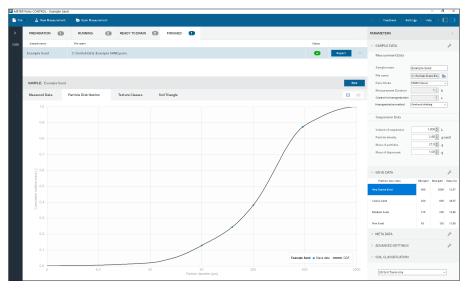


Figure A.14 Example sand particle distribution results

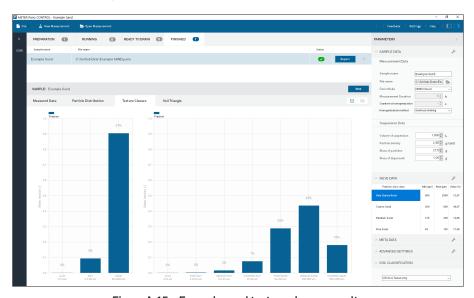


Figure A.15 Example sand texture classes results

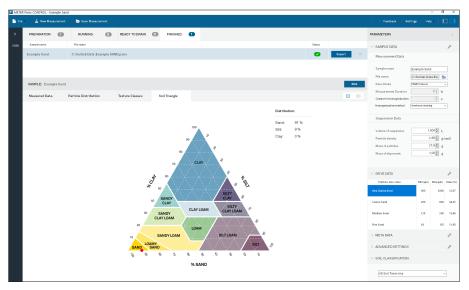


Figure A.16 Example sand soil triangle results

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