PARIO
USER MANUAL

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1. INTRODUCTION

Thank you for choosing PARIO© from METER Group.

Please verify all instrument contents (see 1.3) shipped and appear in good condition.

This "Short Manual" is intended as a quick start-up for users that are familiar with the sedimentation method, and used it so far as pipette method or hydrometer method. Users that are inexperienced in performing sedimentation experiments for particle-size analysis should refer to the respective operation protocols that are described in text-books and method books, and to the extended PARIO© manual (which will be available by summer 2017).

1.1 THEORY

Soil is subdivided in fine soil (< 2000 µm) and gravel (> 2000 µm). Fine soil again is subdivided in sand, silt and loam. The limits of the particle fractions are defined by equivalent diameters of the particles. These values vary depending on national classification systems.

E.g. German classification:

Particle fraction	Sand	Silt	Clay
Equivalent diameter [µm]	2000 - 63	63 - 2	< 2

E.g. American classification:

Particle fraction	Sand	Silt	Clay
Equivalent diameter [µm]	2000 - 50	50 - 2	< 2

PARIO© is an automated system for the particle-size analysis (PSA) for soils and sediments. The particle-size distribution (PSD) of granular materials is obtained by the sedimentation method, which identifies specifically the silt fractions with hydrodynamic diameters between 63 µm (upper size limit) and 2 µm (lower size limit). The clay fraction is calculated by subtracting the externally measured sand fractions and the silt fraction from the total amount of material used in the analysis. Data for sand fractions are provided by a sieve analysis. Preferable, this analysis is obtained independently and before PARIO© analysis from a splitted sample.

1.2 MEASUREMENT PRINCIPLE

PARIO© uses the PSD to derive the particle-size distribution (PSD) from the pressure decrease at a measuring depth in a suspension. The theory of the method is published by Durner et al. (2017). The article is open access and available here. The sedimentation methodology follows the requirements of ISO 11277, 2009; ISO 13317, 2001; ASTM, 2007 and is based on Stokes' law. Stokes' law indicates that particles of spherical shape settle in a suspension with a characteristic velocity which is given by the fluid viscosity,

the density difference between the particle material and the suspension fluid, and the diameter of the particle.

The preparation of the suspension with the particles to be analyzed involves destruction of organic matter and possibly binding agents, and dispersion of the material. This is not described here, because the user does it in exactly the same way as he/she is used to in the traditional methods. First-time users that are inexperienced in performing sedimentation experiments for PSA are referred 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 13317-1, 2001; ASTM, 2007), or to the extended PARIO Manual.

1.3   SCOPE OF DELIVERY AND SYSTEM OVERVIEW

The PARIO© measuring system consists of

- one PARIO© device,
- two glass sedimentation cylinders,
- one plug,
- one download card for PARIO Control software.

The PARIO© device consists of a pressure sensor at the tip of a shaft, which is connected to a measuring head. The shaft is submerged in a suspension with settling particles. At the side of the pressure transducer is a temperature sensor. The pressure and temperature signals are processed in the measuring head on top of the sedimentation cylinder. The signals are transferred in digital format via an USB connection to a PC. In data sampling mode, one data triple (time, pressure, temperature) is recorded every 10 seconds to the PC.

2.   INITIAL OPERATIONS

2.1   INSTRUCTIONS FOR USE

1. The device must only be used in laboratories.
2. The device must be placed on a horizontal, vibration-free and solid surface.
3. Avoid direct sunlight exposure at any time!
4. Avoid placing below air-conditioning or a window with falling cold air.
5. Samples, device and suspension fluid must be equilibrated at room temperature. Room temperature should be constant at ± 1.5 °C. If possible, use a temperature-controlled room or place sedimentation cylinders in a temperature-controlled water bath.

IMPORTANT: Points 2, 3, and 4 are all strictly required to avoid uncontrolled temperature fluctuations. Temperature fluctuations will strongly affect the accuracy of measurement results!

2.2   INSTALLING THE PARIO SOFTWARE AND HARDWARE

PARIO© requires an installation of the software PARIO Control and the PARIO© USB driver. This installation package can be downloaded under *www.metergroup.com/downloads/pario*

Install the software by selecting the "setup.exe" and following the installation routine that guides you through the process. During this installation process the PARIO Control Software and the PARIO© USB driver will be installed.
The PARIO© instrument must be connected to a computer with a Windows (at least Windows 7) operating system, and the PARIO Control software during the whole measurement period.
Multiple PARIO©s can be used simultaneously by connecting them to an USB hub (min. USB 2.0, 500 mA per port).

2.3   BASIC SETTINGS

With the PARIO© instrument(s) connected to the PC, start the PARIO Control software by double clicking on the respective icon. Upon connection to the PC, the LED rings of the PARIO©s will shortly flash in white color. Connected PARIO©s will show a permanent white light.
The PARIO Control software will pop up in a new window. In this window, first open the menu point "settings" at the top right.

In the settings window, you can set various parameters, which will be applicable for all subsequent individual measurements. The settings are categorized into three registries: ‹General›, ‹Default Parameters›, ‹Sieve Classes›.

Duration and particle density are given as default values but can be changed for individual configuration of the measurement.
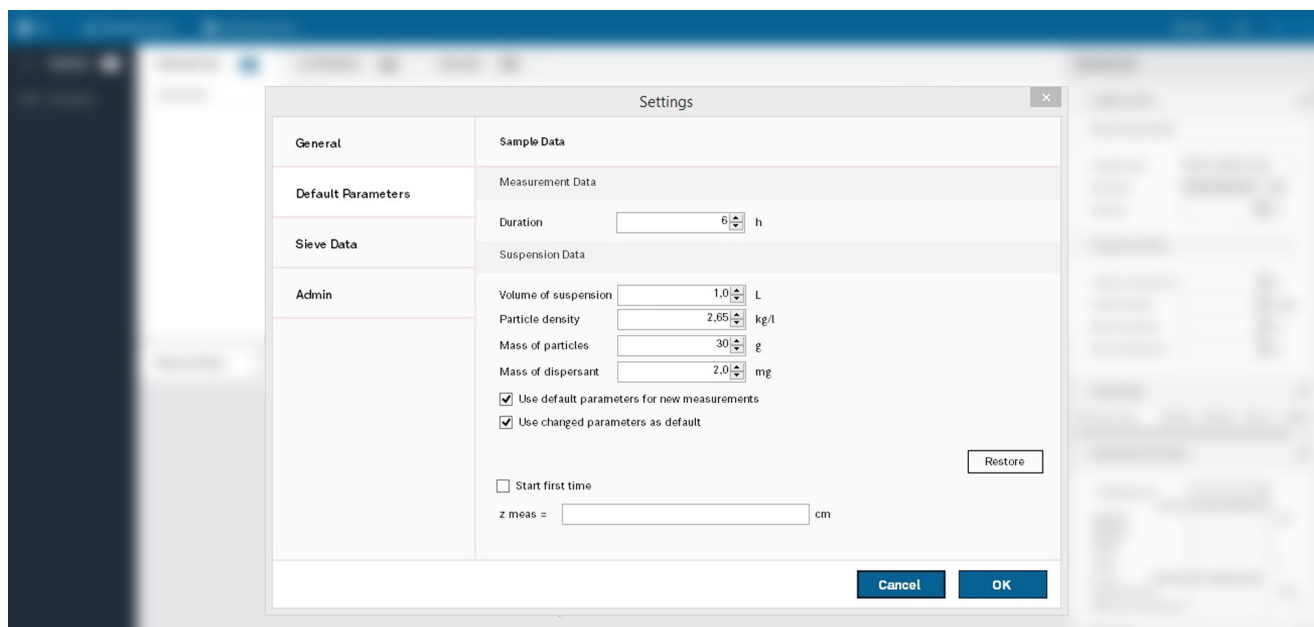
2.4   ADVANCED SETTINGS

IMPORTANT: These are settings which should not be changes by users, who are not very experienced.

relWeight P	Default: 1	Relative weight of pressure data class in object function.
relWeight F	Default: 1	Relative weight of sieve data class in object function.

relWeight F — Explanation:

In determining the particle-size distribution by inverse modeling, the 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 thus data with different units, magnitude and number. PARIO© weights the individual data by an internal weighing scheme. The user can modify this default weighing by increasing the importance of pressure data (relWeight P > 1) or the importance of the sand fraction data from sieving (relWeight F > 1). 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.

FFVER	Default: 2	Index for type of FreeForm function.

FFVER — Explanation:

FFVER determines the cumulative particle size distribution function with a free form spline function with a prescribed number of support points. FFVER = 1 uses less support points than FFVER = 2. As a result, FFVER = 1 leads to a faster fitting time but ha less flexibility to describe the data.

TCRIT	Default 60 s	Time of first data point to be used in the evaluation of the pressure time series. Explanation: PARIO© records data normally from 20 s on. This early pressure decrease is affected by particles of all sizes, including sand particles. Using these earliest data thus contains information from sand particles, which fall very quickly, possibly in a turbulent manner, which cannot be described by Stokes law. Thus, if sand is in the sample to a considerable part, the very first pressure data should not be used in the PARIO© analysis.
Liquid expansion	Default: 1.3 Pa/°C	Explanation: The temperature during a sedimentation analysis should be kept a constant as possible. However, under some circumstances, moderate temperature drift might occur in a lab environment, which is recorded by PARIO©. The PARIO© pressure sensor itself is temperate compensated, but different thermal expansion of liquid and solid parts of the experimental setup will cause a temperature drift of the pressure signals in a certain depth. To compensate for thermal expansion, PARIO© uses an experimentally determined value of 1.3 Pa/°C (i.e., pressure head change of 0.13 mm/°C).

2.5   LED INDICATORS

The connected PARIO©s can be individually and simultaneously used to perform particle-size analysis experiments. The status of the instrument is indicated by the LED light ring:

White	flashing, 3x	PARIO© has been detected by the computer
White	permanent	PARIO© has been detected by the PARIO Control Software. The device can be configured for measurement.
White	flashing	PARIO© is ready for measurement
Blue	pulsing	PARIO© measurement is in progress
Blue	permanent	PARIO© measurement is finished
Red	permanent	during firmware update
Red	flashing	Hardware error. Please contact vendor.

If the device does not flash white 3 times when it is connected to the computer please skip to point 5.3 "Trouble shooting".

3. PERFORMING A PARIO© MEASUREMENT

3.1 SOIL SAMPLE PREPARATION

Prepare the sedimentation experiment in a sedimentation cylinder in exactly the same way as usually done for a pipette or hydrometer analysis. Preparation of the material follows the user's needs.

Typical steps encompass
- sieving to 2 mm,
- destruction of kitting substances,
- destruction of organic carbon,
- dispersion of the material,
- transfer of the dispersed slurry into a standard sedimentation cylinder (provided as part of the PARIO© system by METER).

The amount of sediment to be used in a PARIO© experiment should be in the range 25 g (material without sand) up to 50 g (material with much sand). If nothing is known about sand content, we recommend to use 30 g dry weight. Be precise with respect to the dry weight of the sedimenting material – the accuracy of the analysis depends on it!
Fill one cylinder with 1 liter of distilled room tempered water and cap it.
Give the soil sample into the second cylinder and use distilled room tempered water to fill it up to the one liter mark.

3.2   SOFTWARE CONFIGURATION
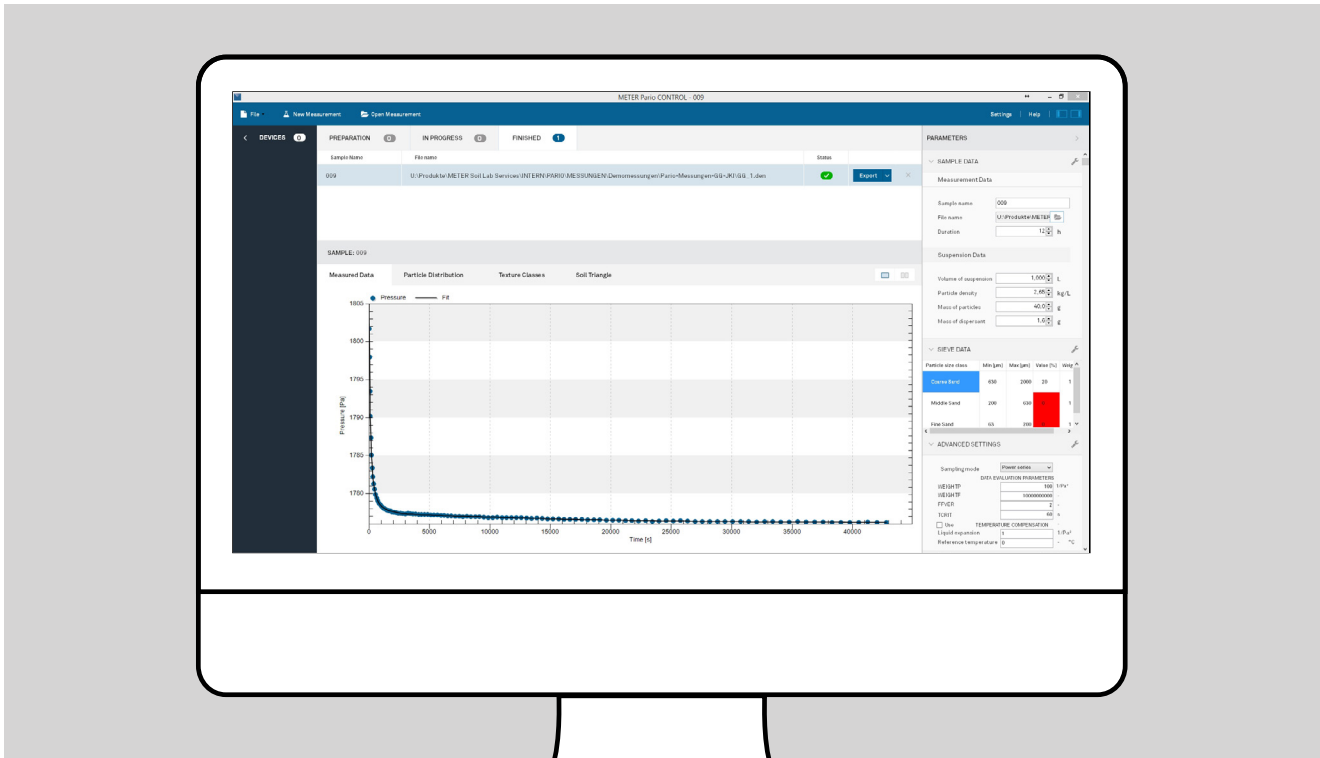
Connect all used PARIO©s to the computer and start the software.
The devices should flash white. If not, reconnect and connect them again.

Select "New measurement".

Following parameters are defaulted and can be changed manually if desired:
- "Duration": 6 h
- "Particle density": 2.65 g/cm^3

Following parameters have to be entered:
- "Sample Name" -> file name will be automatically generated and can be changed manually
- "Volume of suspension"
- "Mass of particles"
- "Mass of dispersant"

The values you have entered are saved as default settings for all following measurements. Still they can be changed manually in the parameters or directly in the default settings at any time. They can also be deactivated under "Settings", "Use default parameters for new measurement".

When all necessary parameters are entered the status changes to green, a check mark appears and the "start" button is selectable.
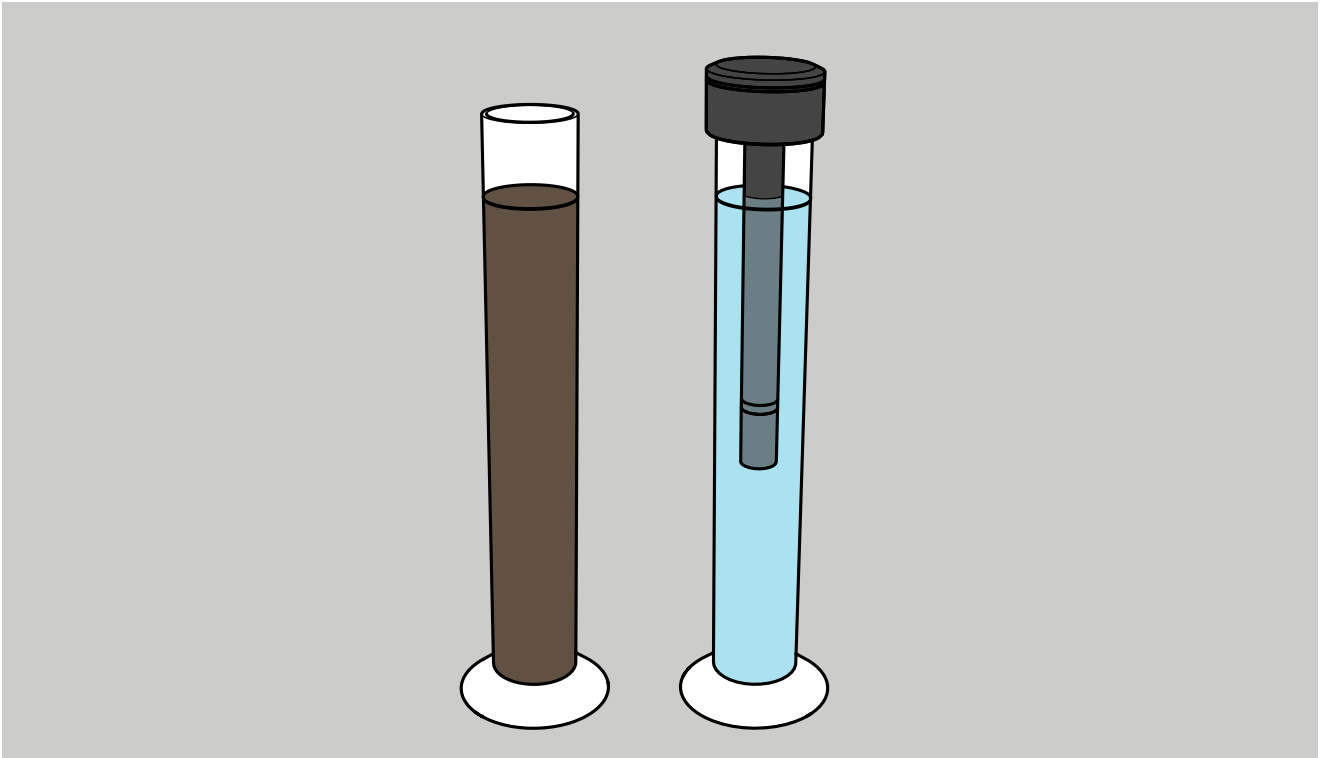
For performing a sedimentation analysis with PARIO© you need to place one cylinder with the prepared soil sample and the other one with distilled room tempered water on a plane surface. Insert the PARIO© in the distilled water for temperature equilibration and connect it to the computer.

NOTE: Be sure that the suspension as well as the part of the device which is inserted in the suspension have the same temperature.

3.4   STARTING A MEASUREMENT
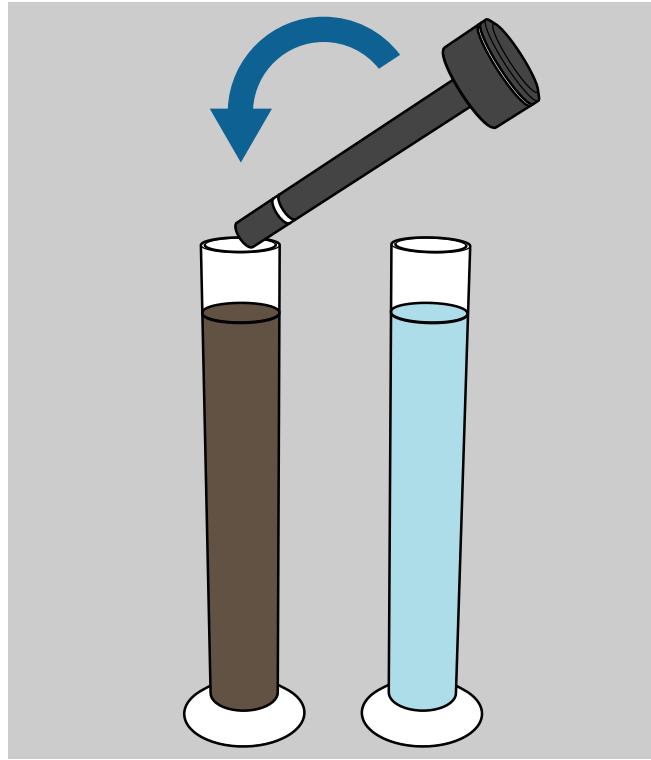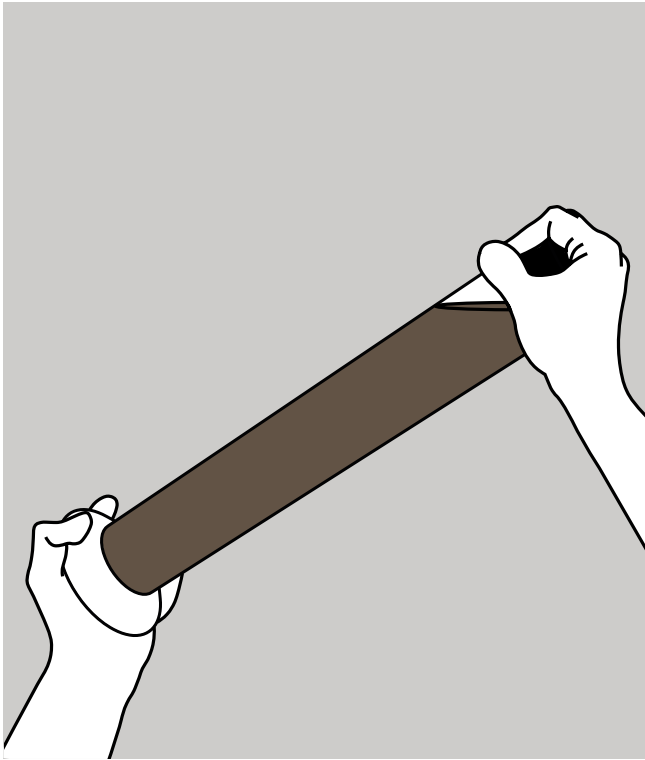
To start a measurement, press the "start" button for the selected sample in the "preparation window" of PARIO Control.

This initializes the Countdown for mixing. The countdown should be set to 60 sec minimum time and can be configured in the general settings. During the countdown, mix the suspension thoroughly. This can be done either by continued upside-down turning of the cylinder (which must be covered with the included water tight plug) or by 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 initial suspension because the whole measurement depends on it. Any error in the homogenization will propagate through the analysis and give wrong values.
>
> **IMPORTANT**: Take particular care before the start of the mixing that no slurry is stuck to the base of the cylinder. To bring this into suspension, use very vigorous shaking and/or stirring and check it visually. This must be done before the countdown starts.

Stop the mixing exactly when the countdown reaches 0 seconds. In the case of overhead shaking, place the sedimentation cylinder now at its final position for the measurement. Now move the PARIO© from its parking position to the measuring cylinder. Before inserting the PARIO© into the suspension, bring it for a moment to a horizontal orientation (approx. 45°) which activates the automatic device detection in PARIO Control. Then insert it into the suspension and make sure that the rim of the sedimentation cylinder is in the nut of the PARIO©. The time from the end of the mixing until the final insertion of the PARIO© should not exceed 30 s.

3.5   MEASUREMENT PROCESS
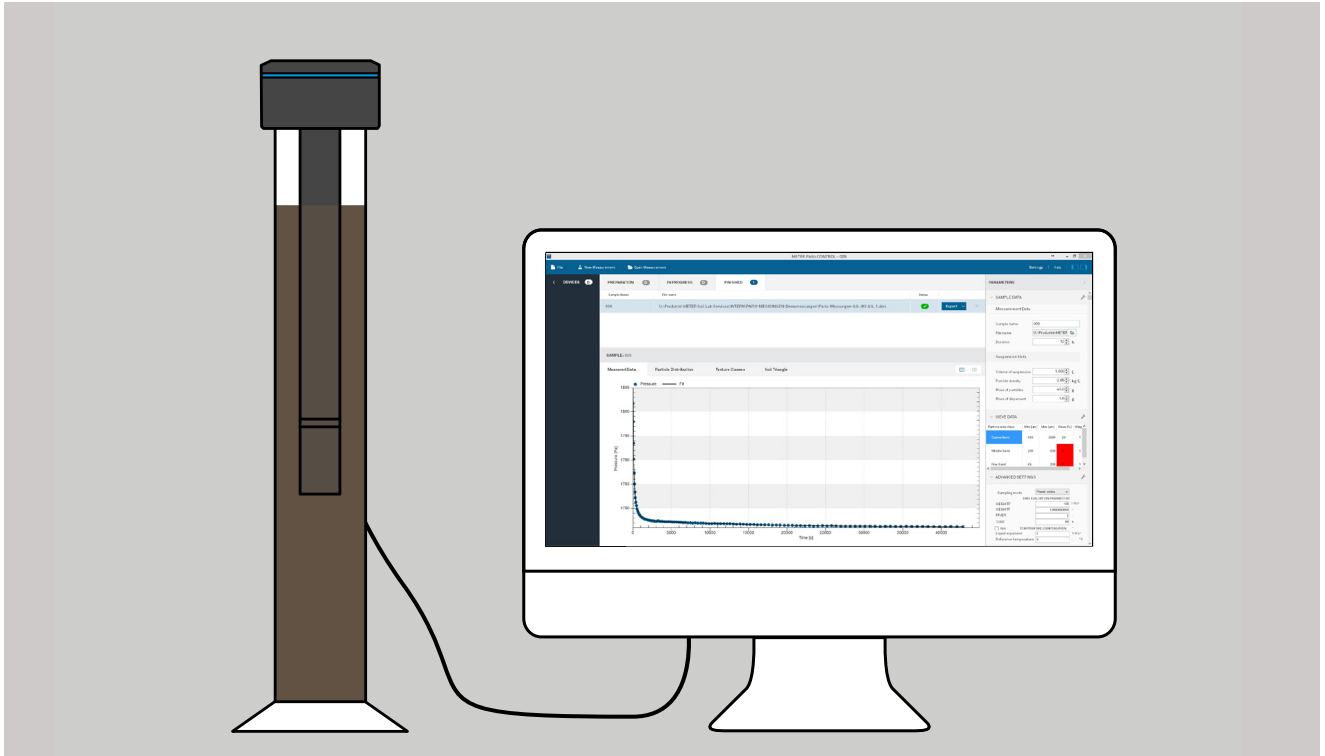
From now on, PARIO Control will record a measurement data triple (time, pressure, temperature) each 10 sec and depicts the recorded data graphically. The status line of the running measurement is moved automatically from the list "Preparation" to the tab "In Progress".

For the operator, there is nothing to do now until the end of the measurement.

If multiple PARIO©s are used, the operator can now proceed with mixing the next suspension. Switching windows in the PARIO© software will not affect the running measurements.

3.6   END OF MEASUREMENT
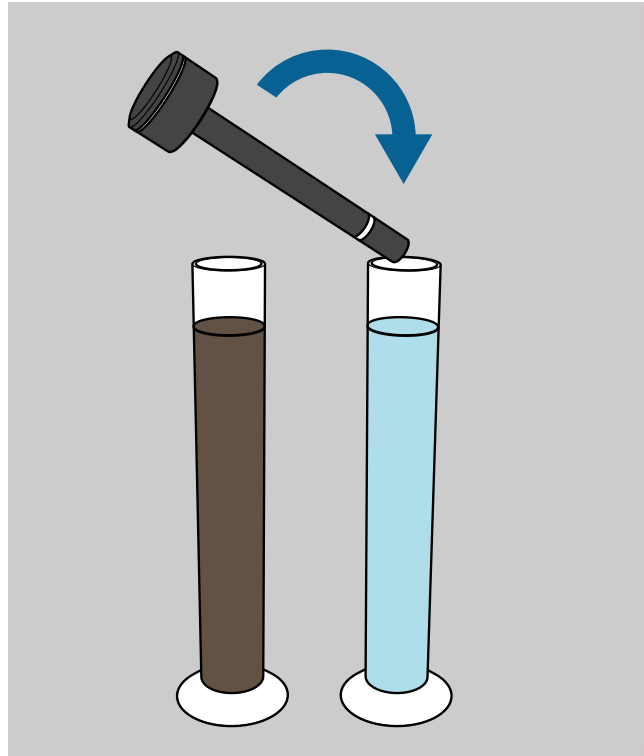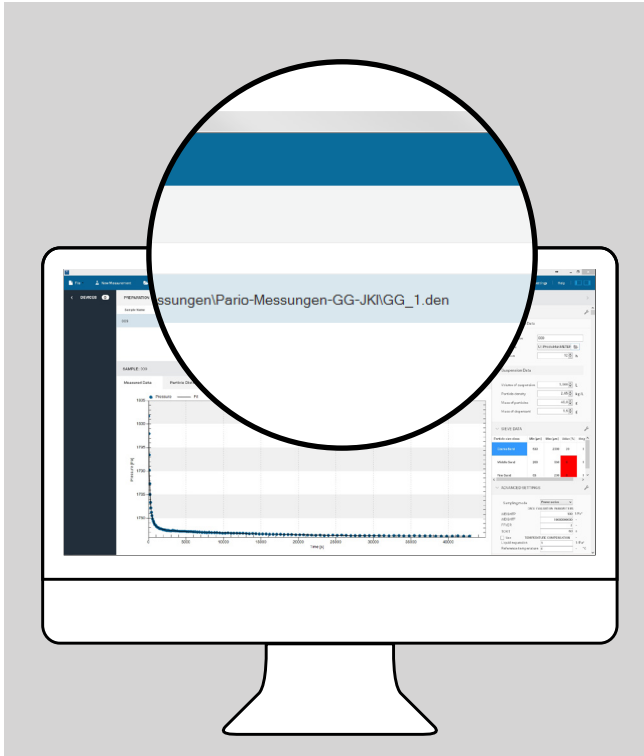
A measurement finishes automatically when the entered duration is over. Alternatively, a user can stop a measurement any time by clicking on the respective button in the PARIO© software.

When the measurement is stopped, it is listed in the tab "Finished".

For short time storage (next measurement within a couple of days), just remove the PARIO© from the suspension and place it again in the cylinder with distilled water. If necessary, rinse the shaft of the PARIO© with a soft jet of water. Also, you might clean the shaft with a soft cloth. For long term storage please mind outline point 5.1.

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

3.7   DATA EVALUATION AND EXPORT

The evaluation of the measurement data follows the theory outlines in section 1. It is performed at any time during the measurement or finally at the end after data recording stopped.

PARIO Control provides for each measurement four output screens:

MEASURED DATA

Shows the recorded data of pressure and/or temperature versus time. Time axis can be toggled between linear and logarithmic. Data can be shown either all, or in a reduced amount ("logarithmic", "power").
After fitting the pressure decrease, additionally the fitted line is shown in superposition to the data.
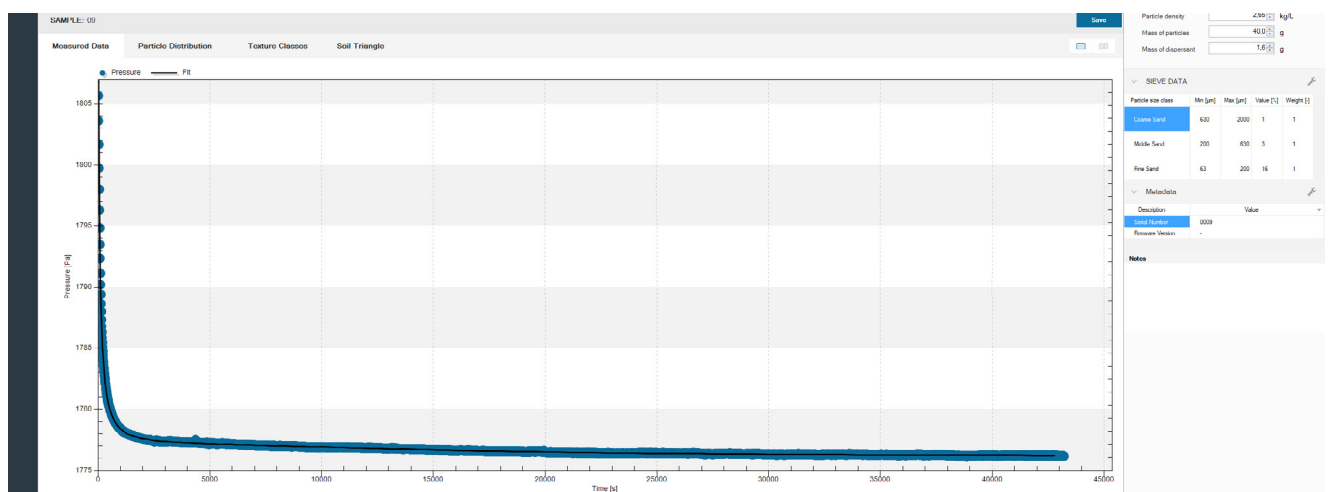
PARTICLE DISTRIBUTION

This window shows the result of the analysis in form of the cumulative PSD, which is available after fitting the data. IMPORTANT: The total cumulative PSD is only correct, if the proper sieve fractions have been inserted to the respective data window ahead of the fitting.
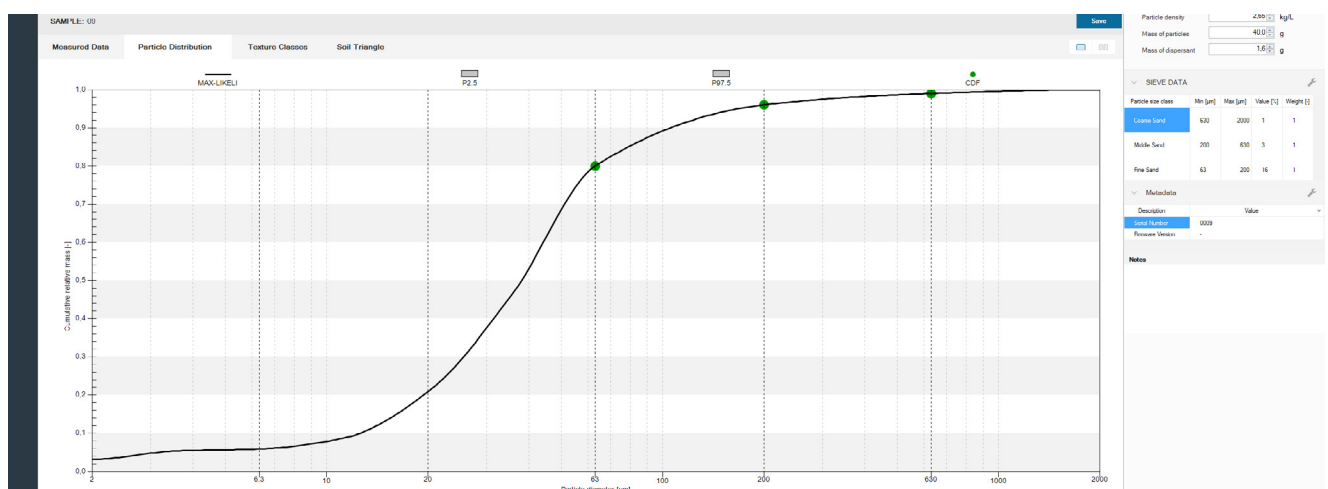
TEXTURE CLASS
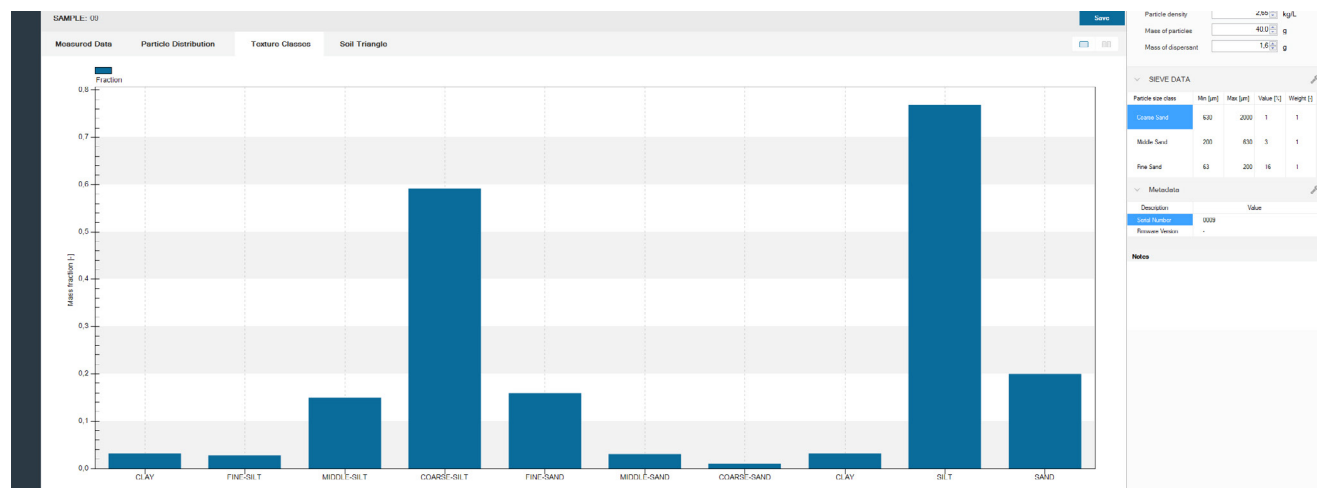
SOIL TRIANGLE
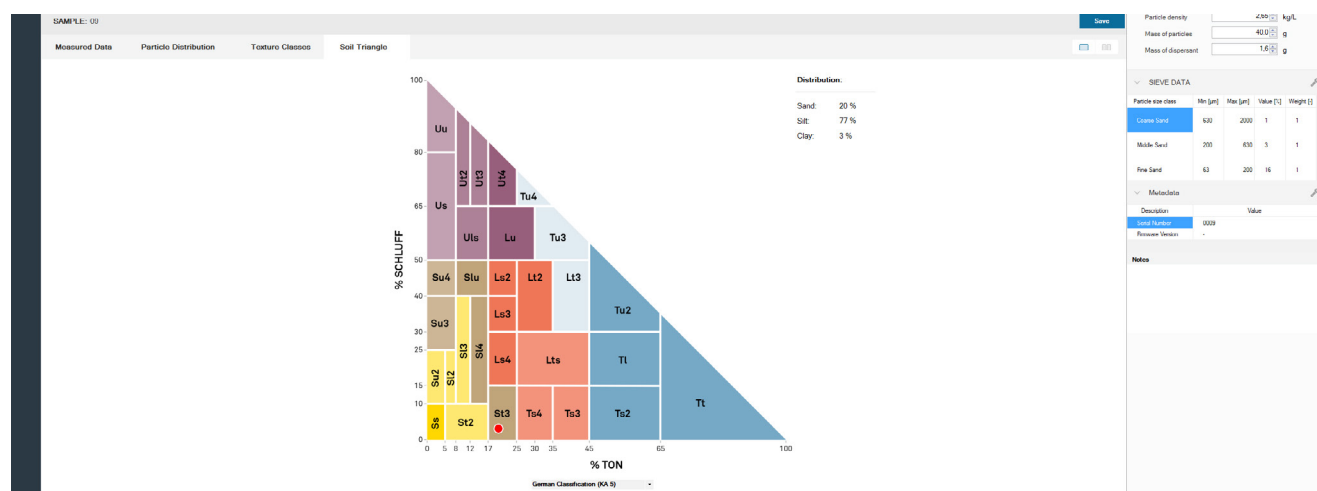
The recorded measurement data and test results are automatically saved and stored after each measurement run on the specified directory in the specified files. File names are composed by the user-specified main name and a generic file name ending (".den") in a binary format.

These can be exported by spreadsheet programs such as MS-Excel©. Diagrams can be exported as an image in the .jpg format.

4.   TYPICAL MEASUREMENT RESULTS

4.1   EXAMPLE SILT LOAM

Typical measurement of silt loam sedimentation.
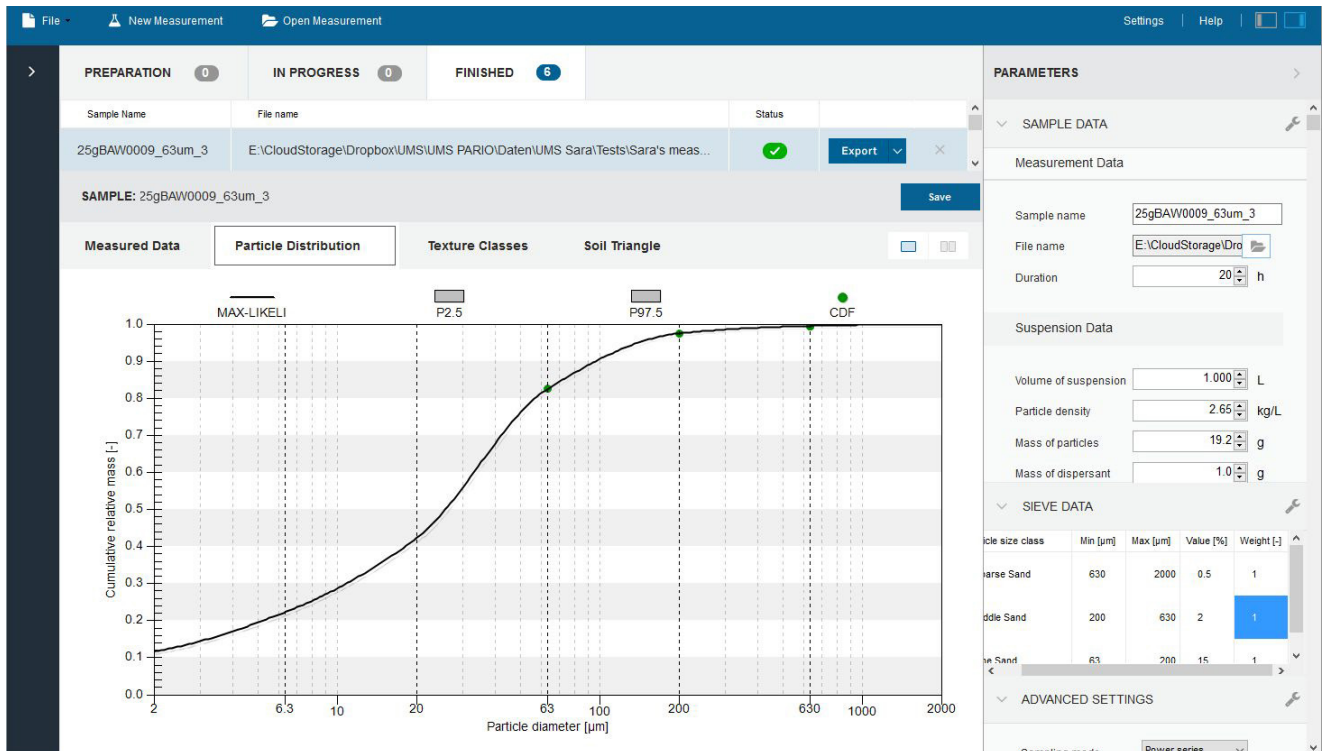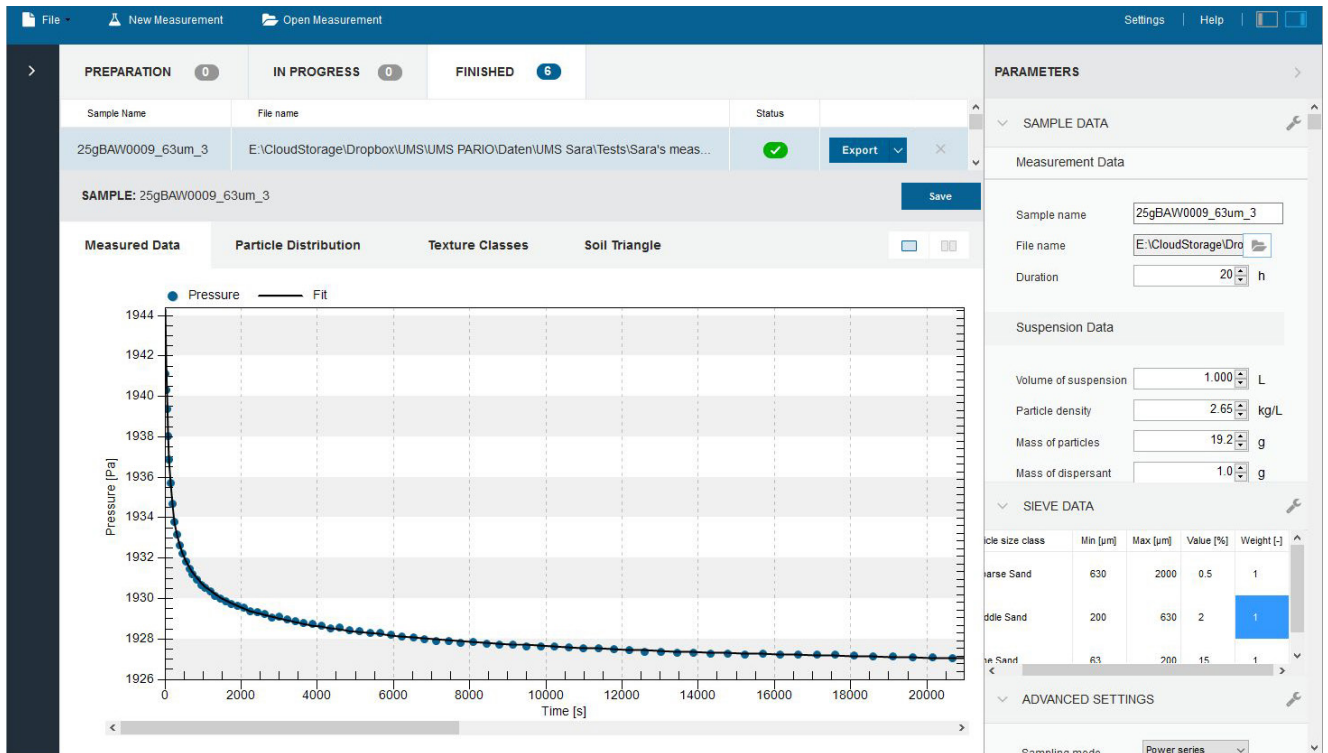
5.   MAINTENANCE AND SERVICE

5.1   STORAGE

Short term storage (few days)
The pressure transducer of PARIO© is ultra-sensitive. Thus, it is strongly recommended to have the PARIO© placed at all times in a sedimentation cylinder that contains one liter volume of degassed distilled water, equilibrated to the room temperature.

Long term storage
If the PARIO© system is not used for a longer time period, it is recommended to clear all parts of the device with water in order to prevent the formation of algae. Then store the device in a dry state.

5.2   CLEANING

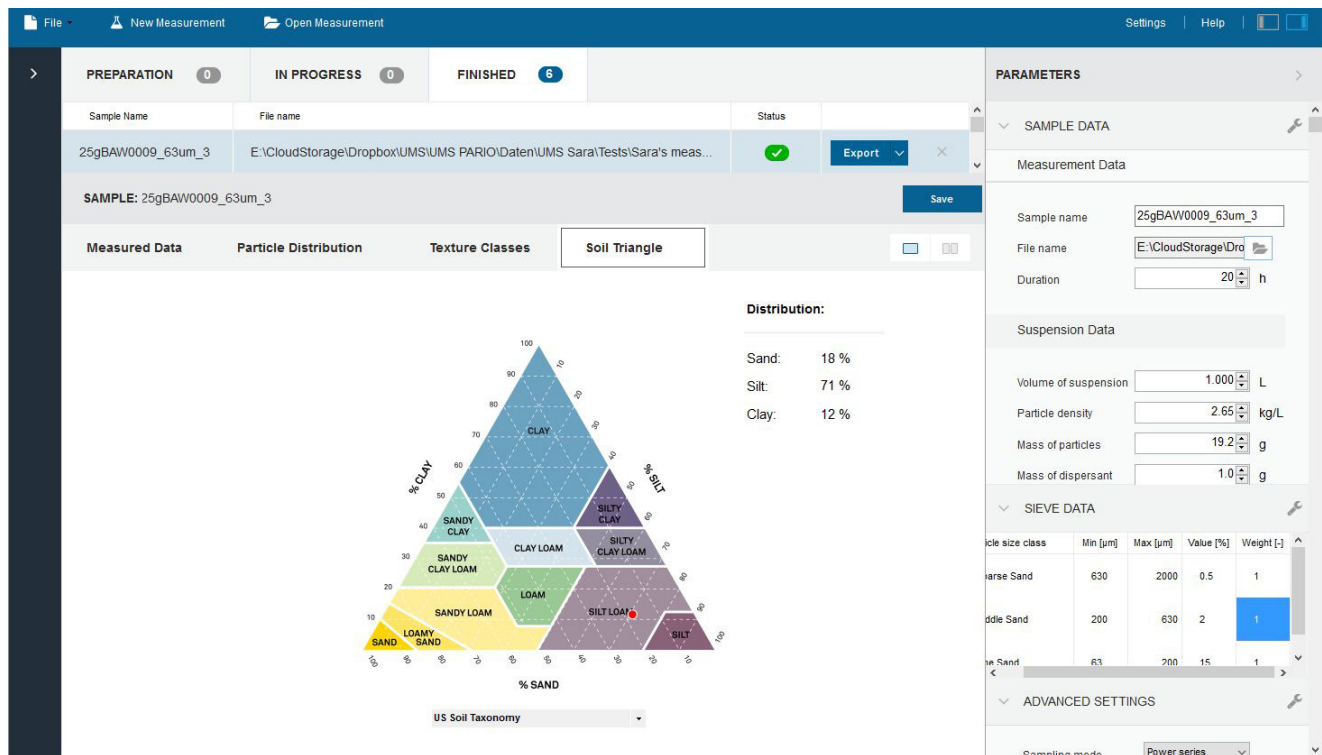The exterior of the PARIO© can be wiped with a damp cloth. Avoid drying of pools of water, since this will lead to salt precipitates. If fine soil and sand grains are deposited in socket and sediment into the base at the end of a measuring campaign, clean the entire base under a gentle spray of water. Take care not to hurt the pressure transducer, by avoiding contact with hard sharp objects or a strong water jet.

5.3   TROUBLE SHOOTING

PROBLEM	CAUSE / SOLUTION
No white flashing 3 times when connected. PC does not recognize the device	1. PARIO© is not connected to the computer. 2. Disconnect the USB and connect it again. 3. Check the PC's Device Manager to see if PARIO© USB driver is installed correctly. Install the PARIO© USB driver again if necessary. 4. Start the PARIO Control software again. 5. USB hub has not enough power
Start of measurement has not been recognized automatically by inserting the device into the sample cylinder.	1. Close the countdown window 2. Place the device back into the cylinder with distilled water 3. Press "Start" and homogenize the sample again.
Computer goes to standby mode and measurement was interrupted.	1. Deactivate the Standby mode in the settings of your computer and start the measurement again.

5.4 CUSTOMER SUPPORT

Have a question or problem?

Our support team can help. We manufacture, test, calibrate, and repair every instrument in house. Our scientists and technicians use the instruments every day in our product testing lab. No matter what your question is, we have someone who can help you answer it.

Contact our customer service representatives with questions, problems, or feedback:
support.environment@metergroup.com
sales.environment@metergroup.com

Phone GER: +49 126652-0
Phone USA: +1 509.332.5600

If contacting METER by email or fax, please include: name, address, phone, fax number, instrument serial number, and a description of the problem.
For instruments purchased through a distributor, please contact them for assistance.

6. FACTS & DATA

6.1 TECHNICAL SPECIFICATIONS

Range of Particle Sizes	63 µm to 1 µm
Approximate error in mass fraction detection	+/- 1.5 %
Accuracy of pressure measurement	0.1 Pa
Typical particle mass	25 to 40 g / 1 litre suspension
Typical duration of measurement	6 hours
Measuring interval	10 s
Operating temperature range	15 °C to 35 °C
Max. tolerable temperature change during measurement	3 °C
Volume of suspension	1000 cm³
Height of sedimentation cylinder	35 cm
Power requirement	1 mW
Warranty	12 months
Required external measurements	Gravimetric sand fractions

6.2 EFFECT OF TEMPERATURE

The viscosity of water is highly temperature dependent. Dynamic viscosity can be approximated by the function $y = 0.0007 T^2 - 0.0531T + 1.764$ ($r^2 = 0.9996$), where T is given in °C. Table 1 lists some characteristic values of the viscosity at various temperatures.

Temperature in °C	5	10	15	20	25
Dynamic viscosity of pure water [mPa s], at 1 bar	1.518	1.306	1.137	1.001	0.894

PARIO Control records the temperature during a run and calculates additionally the mean temperature from it. Settling velocity is calculated for a constant particle density of 2.65 g/cm³ and a viscosity and density of water that is temperature-dependent.

The measurement can also be stopped manually by the operator at any time before the automatic stop is reached.

6.3   EFFECT OF GAS BUBBLES

Gas bubbles may occur when the water in the suspension is not equilibrated with the air at ambient temperature. Gas bubbles at the pressure transducer will affect its reading, and air bubbles somewhere else in the system that stick to liquid/solid interfaces may led to bias in the weight of the water column above the measuring depth. Thus, gas bubbles should be avoided. To avoid gas bubble formation, use slightly degassed water, e.g., by heating the deionized water prior to its use in preparing the suspension.

6.4   ACCESSORIES

PARIO© plug	Order code: 020651
PARIO© glass sedimentation cylinder	Order code: 020650

6.5   TERMS AND CONDITION

PARIO© has a one-year warranty on parts and labor. The warranty is automatically validated upon receipt of the instrument. Fulfillment takes place at Munich, Mettlacher Straße 8.

7. REFERENCES

ASTM (2007):
D422-63 Standard Test Method for Soil Particle-Size Analysis of Soils.

Tan, K. H. (2005):
Soil sampling, preparation, and analysis. CRC press.

ISO 13317-1 (2001):
International Standard "Determination of particle size distribution by gravitational liquid sedimentation methods - Part 1: General principles and guidelines.

ISO 13317-2 (2001):
International Standard "Determination of particle size distribution by gravitational liquid sedimentation methods - Part 2: Fixed pipette method.

Durner, W., S.C. Iden, and G. von Unold (2017):
The integral suspension pressure method (ISP) for precise particle-size analysis by gravitational sedimentation. Water Resources Research, 53, 33-48, doi:10.1002/2016WR019830.

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2.4 Particle-size analysis. In: Methods of soil analysis, Part 4, 255-293.

ASTM D422-63 (2007):
Standard Test Method for Particle-Size Analysis of Soils. Annual Book of ASTM Standards.

DIN 18123 (2011):
Soil, Investigation and Testing- Determination of Grain-Size Distribution (German: ''Baugrund, Untersuchung von Bodenproben – Bestimmung der Korngroeßenverteilung''). German Norm, Beuth Verlag, Berlin.

ISO 11277 (2009):
Soil Quality – Determination of Particle Size Distribution in Mineral Soil Material – Method by Sieving and Sedimentation, Reference Number ISO 11277:2009(E), International Organization for Standardization. Geneva, Switzerland.

ISO 13317-2 (2001):
Determination of Particle Size Distribution by Gravitational Liquid Sedimentation Methods - Part 2: General Principles and Guideline, Reference Number ISO 13317-2:2001(E), International Organization for Standardization. Geneva, Switzerland.

Durner, W.; Iden, S.; von Unold, G (2016): The integral suspension pressure method (ISP) for precise particle-size analysis by gravitational sedimentation. Braunschweig, München.