Bedienungsanleitung

PARTICLE METRIX

Stabino®

Version 2.0

Stabino Control 2.00.23

Particle Metrix GmbH
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The following operating instructions are valid for the charge titration, independent of whether the system is a charge titration system alone or a combined system with NANO-flex size measurement.

For the determination of the particle size distribution with the NANO-flex system a separate operating instruction is delivered.

Version Stabino Control 2.00.23 January 2014

Printed in Germany.

Table of Content

1. Fundamental safety instructions

1.1 Obligations, liability

1.1.1 Respecting instructions in the operation manual

- These Operating Instructions contain important precautions in order to operate the Stabino® in compliance with the pertinent safety regulations. Knowledge of and compliance with these safety precautions and the pertinent safety regulations are basic prerequisites to ensure a safe and trouble-free operation of the Stabino®.
- Moreover the pertinent regulations and prerequisites applying to accident prevention at the place of installation shall be observed.

1.1.2 Obligations of the Stabino® owner

The guaranteed properties and warranty shall only be applicable, if the following points are complied with:

- The Stabino® must be exclusively operated by persons trained by PARTICLE METRIX GmbH or its representative.
- The Stabino®. may only be used in accordance with the regulations.
- All specifications defined by PARTICLE METRIX GmbH with regard to handling the Stabino® must be observed. This term not only refers to operation, but also to maintenance and storage of the device.
- Maintenance and repairs may only be carried out by persons authorised explicitly by PARTICLE METRIX GmbH or its distributor to perform such tasks.
- If any parts of the Stabino® are replaced by parts other than original parts supplied by PARTICLE METRIX GmbH, this shall lead to a direct loss of all claims which can be raised vis-à-vis PARTICLE METRIX GmbH.
- Safety-relevant disturbances must be eliminated immediately and prior to the next operation of the Stabino®.
- If the Stabino® reflects any functional disturbances or visible damages, or if any damage is assumed, e.g. in the event of loose parts within the Stabino®, the device shall be put out of operation.

1.1.3 Warranty and liability

The Stabino® has been manufactured in compliance with the requirements stipulated in DIN EN 61000-6-4, DIN EN 61000-6-4 und DIN EN 61010-2-081; VDE 0411-2-081:2004-07:2004-07 and CE, tested and shipped in a proper condition with regard to safety-engineering requirements.
The owner and user is responsible for ensuring that all precautions listed in these Operating Instructions and the regulations applying to the installation site of the equipment are observed.
Warranty and liability claims shall be excluded in the event of damage to persons or property, if at least one of the following points is applicable:

- Use of the Stabino® contrary to the regulations
- Non-authorised intervention into the housing, damage to the sealing on the housing screws
- Inappropriate installation, operation, maintenance and repair of the Stabino®
- Operation of the Stabino® with defective safety equipment or featuring damages which may affect safety
- Operation of the Stabino® without the prescribed or with non-functional safety installations
- Non-compliance with precautions stated in these Operating Instructions with regard to operation, storage, transport and maintenance of the Stabino®
- Any modification of the equipment is against the delivery conditions.

- Damage as a result of intervention by a foreign substance, acts of God or catastrophes
- Faulty maintenance

If the equipment is sent to PARTICLE METRIX GmbH for repairs, always ensure that the appropriate measuring cell and displacement piston are enclosed in the shipment.
The warranty period is 1 year from the date of delivery.
PARTICLE METRIX GmbH shall not warrant under any circumstances for warranty damage exceeding the purchase price of the equipment.
PARTICLE METRIX GmbH is only obliged within the scope of warranty to replace or repair parts classified as defective within the scope of the examination performed by PARTICLE METRIX GmbH at its own discretion, if these parts were returned within the given time schedule.
Parts which are modified or damaged as a result of improper handling without written approval by PARTICLE METRIX GmbH, shall be exempt from warranty.
Furthermore, PARTICLE METRIX GmbH shall not assume any further obligations in connection with selling the Stabino®.

1.1.4 Exemption from liability

The contents of these Operating Instructions have been verified. It is nevertheless impossible to exclude deviations to the device or errors. PARTICLE METRIX GmbH therefore does not assume any warranty for the correctness of the information provided in these Operating Instructions. If appropriate, modifications will be integrated into a following version.
Technical modifications reserved.

1.2 Use according to regulations

The Stabino® was developed to determine the charge and the isoelectric point in aqueous solutions. Operation shall only be permitted in interior rooms, observing the parameter specified under "Technical Data" in conjunction with the accessories released by PARTICLE METRIX GmbH.
Any deviating use or non-compliance with the Operating Instructions shall be deemed to be a use which is contrary to the agreed terms and shall lead to non-liability on the part of PARTICLE METRIX GmbH with regard to the damages resulting therefrom.

1.3 Use contrary to regulations

The following Stabino® shall be deemed to be use of the equipment contrary to the regulations (misuse):

- Each and any application deviating from the use of the equipment in accordance with the above-mentioned terms
- Non-compliance with instructions and safety precautions stated in these Operating Instructions
- Non-compliance with safety regulations
- Operating the equipment despite safety-relevant disturbances
- Operating the equipment despite functional disorder or visible damage
- Any modification of the equipment as against the delivery status

1.3.1 Comportment in the case of misuse

i) In the case, that reactive, flammable, explosive liquids or substances were filled into the sample cell, the instrument has to be switched off immediately to avoid an inflammation.
ii) In the case that toxic substances were filled into the sample cell, suitable protective clothing has to be carried. The instrument has to be decontaminated before using it again.
iii) In case of electrical damage, the instrument has to be set out of operation and to be secured against reuse. In the simplest situation, the power cord has to be disconnected. A warning sign has to be attached to the instrument.

The same rules apply when cables are damaged (Loss of isolation).

1.4 Qualification of staff

Persons who have been trained by PARTICLE METRIX GmbH or by an authorised representative shall exclusively operate the Stabino®.

1.5 Safety instructions

In handling the Stabino®, the following points must be observed:

- Only trained staff may put the device into operation or operate the equipment.
- The Stabino® instrument is envisaged only for use with aqueous samples.
- In normal operation, splashing of the sample is not expected. In spite of this, a protective cap can be obtained to cover the measurement container. (see chapter 4.3
- The instrument must not be filled with easy to vaporize, flammable or explosive sample material. Ignition sources are not allowed to be used in the next environment of the instrument. It is prohibited to use open flames in the surrounding of the analyzer.
- Only original parts supplied by PARTICLE METRIX GmbH may be used for maintenance or repairs.
- Only trained staff may carry out any maintenance operations, in so far as these are described in these Operating Instructions.
- Any maintenance or repair operations which are not described in these Operating Instructions may only be executed by PARTICLE METRIX GmbH or a service company which has been explicitly authorized to perform such operations.
- Repair work inside the instrument is allowed only for personnel authorized by Particle Metrix. On doing so, the instrument has to be disconnected from the mains by pulling the power cord from the mains socket.

Caution!
Displays information that has to be observed to avoid any possible injuries or damage to the equipment or environment.

Note
This sign displays information drawing your attention to special aspects.

Electrical supply!
The operation of the Stabino instrument is only allowed in connection with the delivered Particle Metrix power supply. Before connecting the power supply with the mains it has to be ensured that the supply voltage complies with the voltage printed on the label of the power supply.

Wearing personal protective cloth
When using chemicals with safety hazard the local safety precaution rules have to be respected.

1.6 Provision of the safety instructions

These Operating Instructions, as well as any regulations applicable at the place of installation according health and safety at work shall be stored in direct vicinity of the equipment and must be observed. If the documents mentioned become illegible for example due to damage, they must be replaced.

1.7 Packaging & Transport

In the case the Stabino® should be transported and installed at alternating locations, the following must be observed:

- For truck, train, sea and air freight or shipment by mail an appropriate sturdy outer packaging is required.

Note
If the device temperature after transportation is more than 10 °C below the ambient temperature at the place of installation, it is necessary to ensure that the device reaches room temperature before putting it into operation.

Caution
For transportation it is necessary to clean the measuring cell and displacing piston before packing into an extra package or, if remaining in the instrument it should be in the locked position.

Caution
It is prohibited to store any further small parts in the Stabino® to avoid damage to the device and the spring-mounted precision contacts.

Caution
Empty the dosage pumps prior to transport. This can be performed with the rinsing program in the measurement menu, by keeping the tubes in air.

2. Product Description

2.1 Basics

2.1.1 Interactions at the interface

Besides Van-der-Waals attraction (**Fig. 1**) particle charge as an electrostatic parameter of the particle interface has a high influence to the behavior of a colloid or nano-particle suspension. Nearly any surface has ions looking out of the surface and producing an electrostatic field (**Fig. 2**). The particles being small are easily interacting with any other interface.

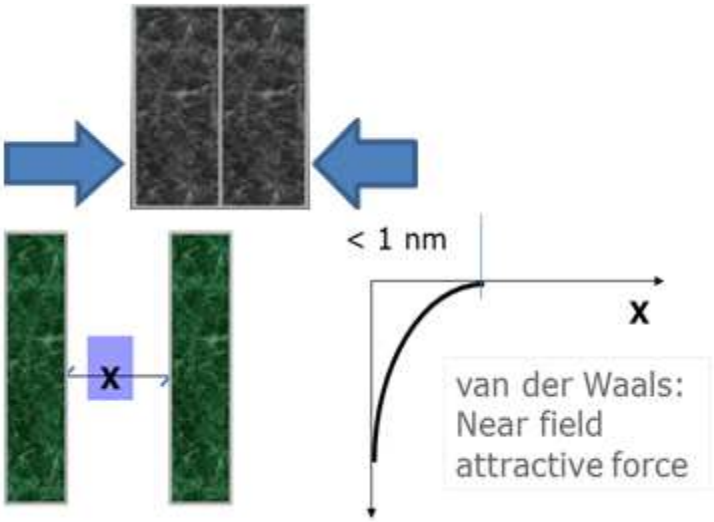


***Fig. 1**: Van-der-Waals force as a short range (< 1 nm) attraction between interfaces. Example flat surface: Left: 2 glass plates near enough to each other to keep attached. Right: Van-der-Waals attractive potential decay.*

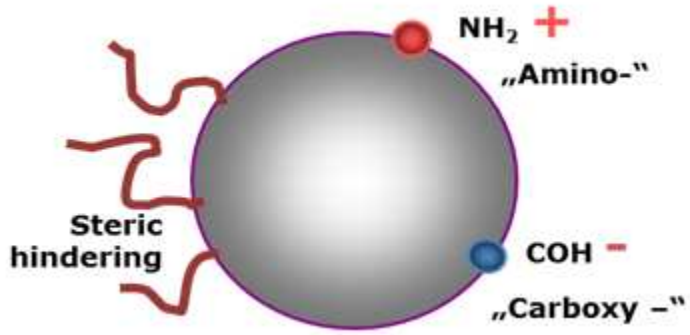


***Fig. 2**: Three different types of end groups at the particle surface: a) Hairy polymers hindering the total approach of others. b) Cationic end group, example amino NH_2+. c) Anionic end group, example carboxy COH-.*

Van-der-Waals attraction and electrostatic forces are the dominant interactions between interfaces. Van der Waals is responsible for the coagulation of dispersion, if no other repulsing effect is present. However, "hairy" polymers "sticking" out from the surface are responsible for "steric hindering", that make a total approach of particles impossible.

Repulsive electrostatic and hairy end groups or both contribute to stability, as shown in **figure 3**.

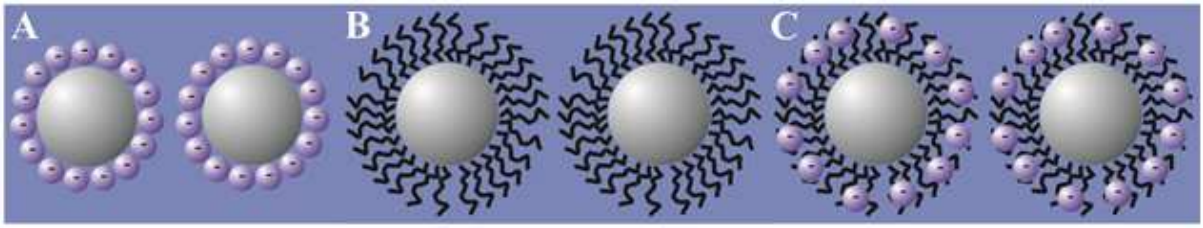


***Fig. 3**: Stabilized particles: A: electrostatically, B: steric, C electrostatically AND steric.*

The following **figure 4** represents the balancing between Van-der-Waals attractive (negative) and electrostatic repulsing (positive) forces. When the electrostatic force is big enough to overcome Van-der-Waals attraction, particles repel or attract each other depending on the amount and polarity of ions sitting on their interface. A stable dispersion is the result of the repulsion between highly charged particles. By adding charged macromolecules (polyelectrolyte additives), the particles gradually accept the character of the additives with respect to the interaction to neighboring interfaces. A dispersing agent is designed to stabilize the dispersion, a flocculant to destabilize it.

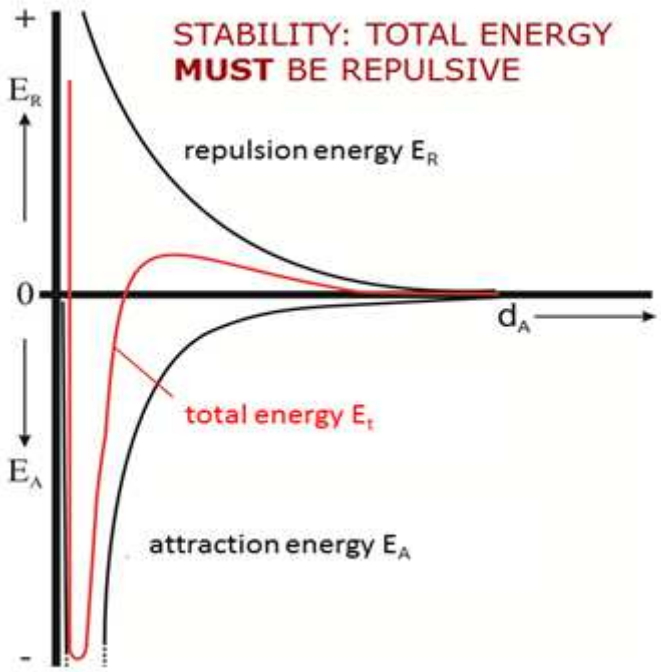


***Fig. 4:** DLVO theory at any interface to a liquid: Competition between the Van-der-Waals (attractive) and electrostatic (repulsive) force. As long as the sum (red curve) is repulsive and higher than the kinetic energy of a possible collision with another surface / particle, the dispersion stays stable.*

The only easily measurable influence out of the three mentioned ones is the electrostatic force. In practice, electrostatic coating is a very frequently applied technique either to stabilize dispersions / colloids or to serve as an intermediate layer between the particle and a functional end group. For formulation work it is important to know the reaction on pH, additives and salts, therefore a "PARTICLE CHARGE **TITRATION**" against these parameters is needed as a **fingerprint**. For quality control, the knowledge of the potential alone is enough.

2.1.2 Double layer and zeta potential

Before we study, how we can measure an electrostatic repulsive energy, let´s have a look inside the double layer. This is important for the understanding of the Stabino® measurement method, which is even easier to understand than LDE Laser Doppler Electrophoresis.

Ions at the surface of particles in a liquid attract mobile small electrolytic ions from the environment. These ions try to shield the charge of the surface, the higher the electrolyte concentration, the quicker is the electrostatic potential decay (**Fig. 5**). As a consequence the thickness of the double layer expressed as the Debye length $1/\kappa$ decreases with the conductivity or ionic strength (**Fig. 6**).

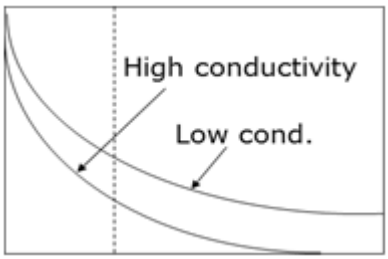


Fig. 5: *Shielding of the particle charge by surrounding ions in the liquid. With increasing ionic strength the double layer cloud, which protects a dispersion from coagulation, becomes thinner.*

Ionic strength [M]	**0.1**	**0.01**	**0.001**	**0.0001**
Debye length 1/k = EDL thickness (1/e) [nm]	**1**	**3**	**10**	**30**

Fig. 6: *According to fig. 5 the extension of the double layer "(Debye length") is decreasing with the square root of ionic strength M. (Origin of the table: A. Dukhin, ISO meeting held April 2012 in Graz).*

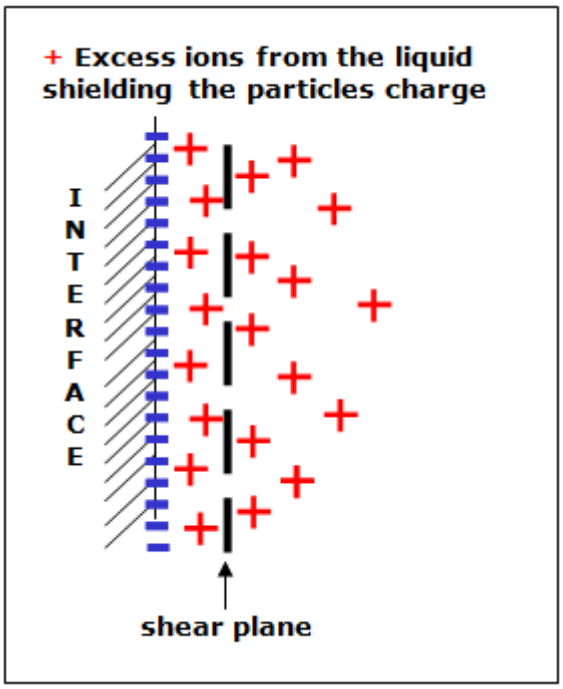


*Fig. 7: Anionic interface with the **cationic counter cloud**, shielding the particle charge to the outside. At the **shear plane** the mobile outer ions can be moved away from the immovably bound inner ions*

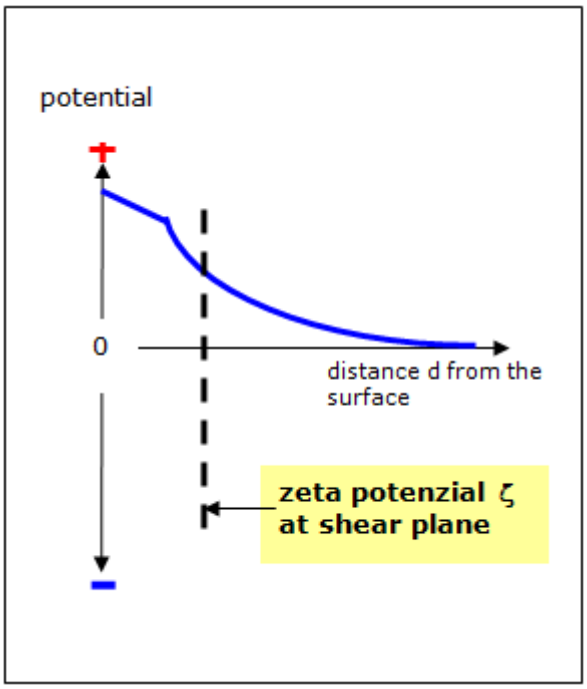


*Fig. 8: Simplified potential decay at the interface. Only the potential appearing during a shearing action is measurable and relevant for any application, the **zeta potential z.***

In the very surrounding of a negatively charged interface positive ions from the liquid "hurry" to the interface to shield its charge to the outside (**Fig. 7**). In the opposite case, when the interface is positively charged, negative ions take over the shielding action. The ionic cloud around the particle interface is called electric double layer (EDL) or diffuse layer. From the charge distribution in the cloud a potential decay can be derived (**Fig. 8**). In practice, a smooth curve as drawn in figure 8 is not likely. As the ions occupy space, the decay may be rather step-wise. The biggest break is at the shear plane. This **shear plane is the only location**, where a potential can be measured with reasonable experimental effort. This potential is called **zeta potential**. In addition, in most processes the size of shearing force is limited. Therefore zeta potential can be seen representative for many practical applications. The next chapter focuses on the measurement method in the Stabino® set-up.

2.2 The measurement of zeta potential

For the measurement of the potential at the shear plane a variety of physical experiments are available. The best known are electrophoresis, streaming potential and ultrasonic set-up´s. The clou is in producing a differential velocity Δv between the interface, no matter how it looks like - flat or like a particle - and the liquid. In electrophoresis Δv is induced with an electric field, in streaming potential with a streaming fluid, in ultrasonic with a sound field. The measured reactions on the excitation are: the velocity of particles in an electrophoresis set-up, a "streaming current or streaming potential" in the streaming fluid set-up, a "vibration current" in an ultrasonic excitation. The measured signal is than calculated as "zeta potential" ZP by using a model, or calibrated to ZP. Zeta potential is the base of common understanding between different methods. It is seen as the parameter of the repulsive energy, which prevents a

dispersion or a colloid from coagulation due to Van-der-Waals collisions between particles. In practice, like in comparing studies, a conversion of the original signal into zeta potential is often not necessary.

2.2.1 Stabino® measurement principle

It is well known from an emptied milk glass that small particles keep attached to the glass wall. That is the basis of understanding for what happens in the measurement cell of the Stabino®. We go back to figure 7, where a heavily charged anionic interface is shown. Take PTFE as a sample cell and fill it with water. The amount of anionic charges on the wall is marginable (**fig. 9 left**). The small amount of charges plus the isoelectric point of the charges can be measured with Stabino®, by the way. Bringing an anionic colloid or dispersion into the cell, the wall is immediately coated with a lot of negative particles (**fig. 9 right**). The same happens with cationic particles, off course.

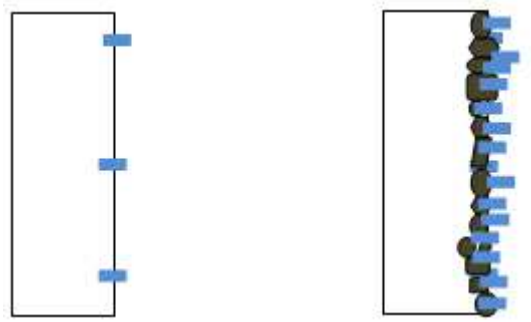


Fig. 9: *Left: PTFE surface in water showing only a few anionic charges. Right: The same surface in an anionic dispersion / colloid. A lot of negative particles are attached. This new "particle interface layer" creates a double layer, illustrated in fig. 10.*

For clarity, only the PTFE wall interface is depicted without illustrating the dispersion. Importantly, by adhering to the wall, the particles are immobilized. Therefore an induced streaming fluid along the wall is able to move the mobile (cationic in the example) excess charges along the wall. An oscillating fluid as in Stabino® creates an oscillating „streaming current or streaming potential"(**Fig. 10**).

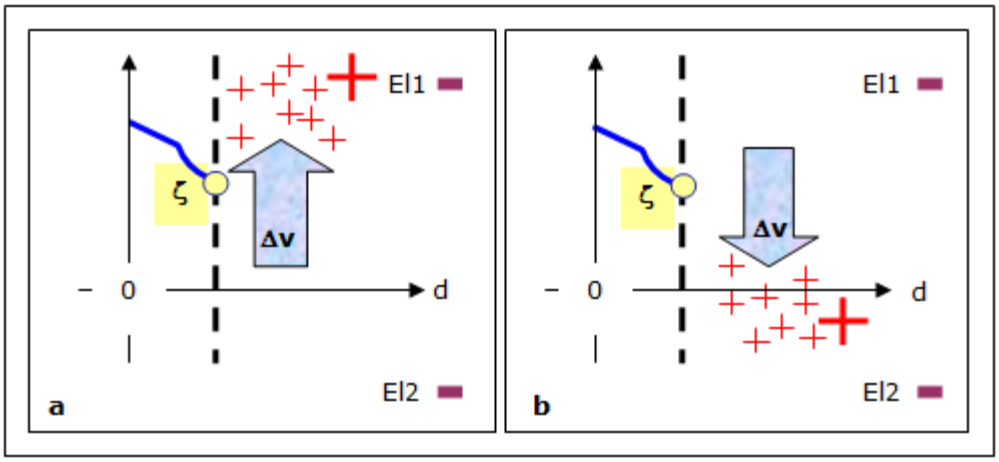


Fig.10: *a) By inducing a fluid stream along the charged wall, excess ions are sheared away from the coated wall creating a streaming potential at the electrodes El1 and El2. The potential can be calibrated as zeta potential. b): The polarity of the potential changes as the direction of the streaming fluid changes. In Stabino®, the fluid is oscillating.*

To complete the picture, on the coarse particle end, from a few µm upwards, particles do not adhere to the wall any more. For these particles the displacement of the ion cloud originates from inertia between the heavy particle and the fluid. A principal chart (**fig. 11**) shows the hand-over of the signal contribution from adhesion to inertia.

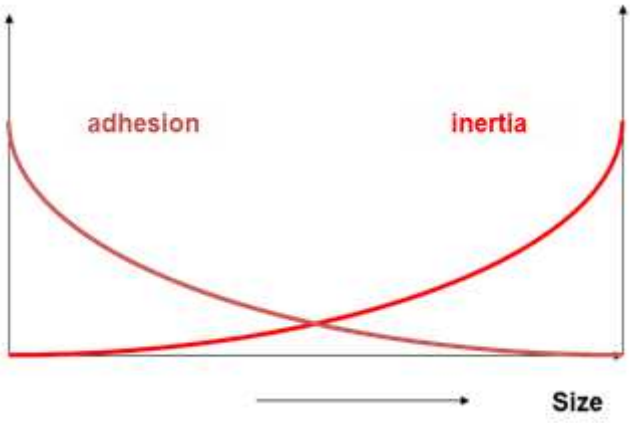


***Fig. 11**: Origin of the streaming potential signal for small and large particles. The low end is at sub-µm, the high end at ~ 300 µm. Cross-over between adhesion and inertia at ~ 5 µm.*

As Stabino® reacts to macromolecules as well as to big particles, it offers the largest size range for particle charge analysis; 0.3 µm – 300 µm. Depending on the sample, the allowable concentration may be between 0.001 and 40 % v/v, which represents an imminently high concentration range. Therefore, Stabino® offers the widest scope of applications.

2.2.2 Stabino® lay-out

The central element in Stabino® is the measurement cell made from Teflon and the oscillating piston in the middle producing an oscillating fluid in the gap between cylinder and piston (**fig. 12**). The narrower the gap at constant motor frequency, the higher is

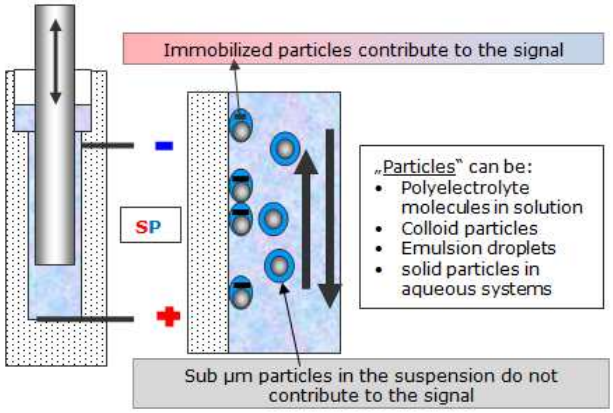


***Fig. 12:** Left: Stabino® Teflon cylinder with oscillating piston and 2 electrodes picking up the streaming potential SP. Right: Gap between the cylinder and the piston showing particles attached to the wall and those, which are in the middle of the fluid. Immobilized particles show ion cloud displacement, "free" particles have negligible cloud polarization. As a consequence, the signal is produced only by the particles adsorbed to the wall. But they represent the charge on all particle´s interface.*

the fluid velocity and the higher the measured potential. The gap can be adjusted to optimum signal to noise, conductivity and viscosity. In the Stabino® design, the driving motor is kept at constant load. Therefore, the oscillation frequency is constant. Depending on the calibration procedure the measurement signal is displayed either as streaming potential or zeta potential (see Chapter 7. "Calibration"). As the measurement is electrical, the signal is instant, one of the greatest advantages of the method, making the experiment rapid. The smaller the particles, the easier they attach to the wall giving rise to an excellent sensitivity. As an example, polyelectrolytes or colloids from sub-nm up to 10 nm show an excellent signal to noise ratio. In this size region all other methods suffer from sensitivity or even worse, are not giving any signal.
The moving of the piston at the same time keeps the dispersion homogeneous and helps to mix-in a titrant solution in seconds. This is shown in **figure 13**. Therefore titrations are extremely effective. It is essential in formulation work to know how the particle reacts on a specific environment: What happens, when the pH or the electrolytic environment changes? What quantity is needed to coat a particle effectively with a certain polyelectrolyte? Stabino® gives always an answer.

The instrument is designed for efficient particle charge titration (**Fig. 14**). Two precision pumps are integrated to deliver either pH solutions, salt or polyelectrolyte solutions to the sample in well-defined dosing steps (see chapter 4, measurement).

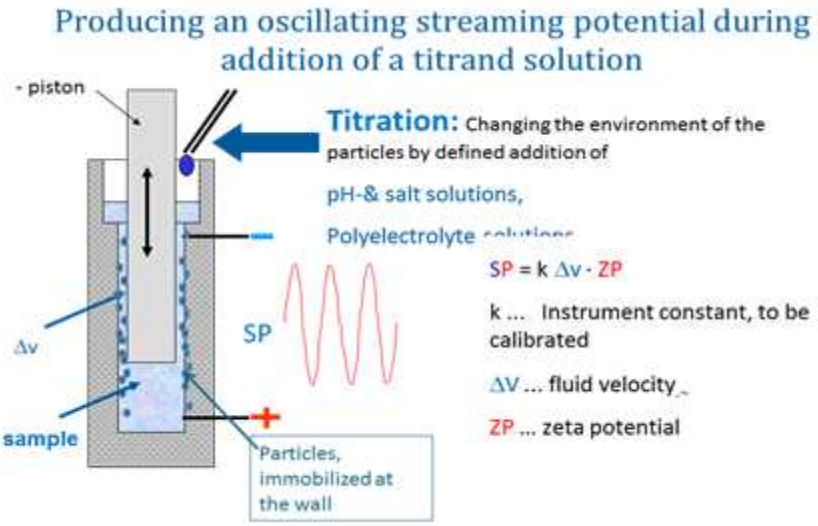


***Fig. 13**: Measurement equipment with Teflon sample cylinder and piston, 2 electrodes at which the oscillating streaming potential signal is picked up. The signal is generated in a second. Important: The piston homogenizes the added titrant solution with the sample in seconds, the reason why a titration with Stabino® is so efficient.*

After introducing 10 mL of sample, the measurement of potential is immediate. A titration is running automatically in the way as a pre-programmed type of titration is selected. One titration lasts a few minutes only.

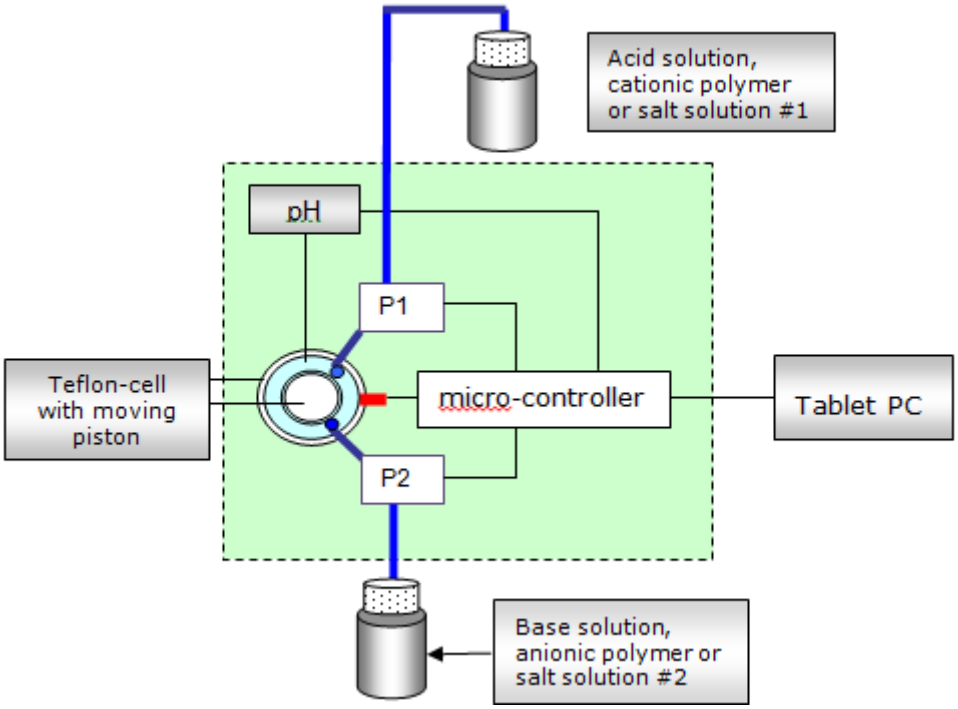


***Fig. 14**: Basic set-up of Stabino® with two integrated dosing pumps and 2 reservoirs for the automatic titration. The sample cylinder has one notch for a pH probe and one for a 180° DLS size probe of an optional NANO-flex instrument (not shown).*

2.2.3 Various kinds of titrations

Fig. 15 shows the most frequently applied titrations. Y-axis represents the potential. pH, consumption [mL] or total charge [C], conductivity [µS/cm] or salt concentration, and time in the case of a kinetics are displayed on the x-axis. The end point of a titration can be programmed freely. However most of the time it is the neutral point (0 mV). The optimum titration result is between 1.5 and 7 mL. To achieve this, the concentrations of the sample and of the titrant solution have to be tuned to each other. The software described in chapter 4 offers easy programming. All measurement parameters are stored. Specific parameters and results may be selected by the user to have a custom styled presentation and report. Exporting data and recalculation is also offered.

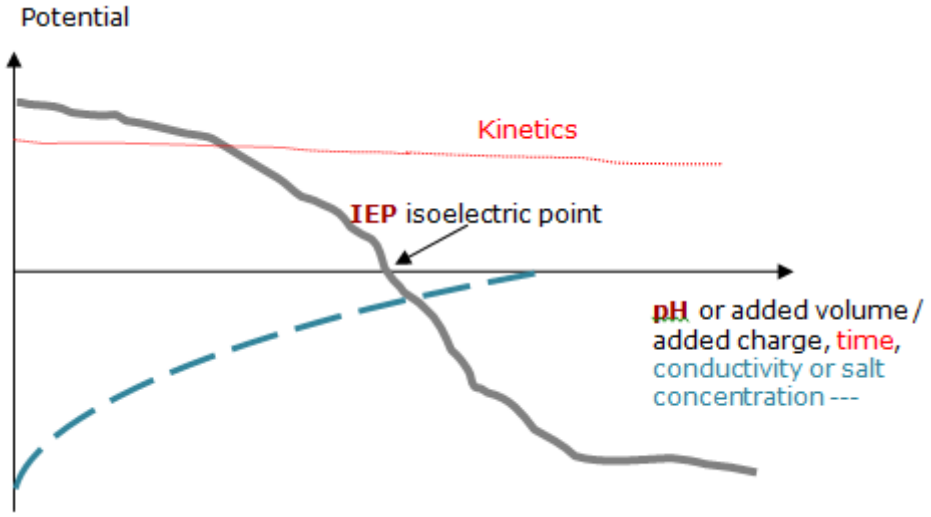


Fig. 15: *Various kinds of titrations*

2.3 Typical type of applications

2.3.1 Titration to the neutral point

- Near the neutral point, the dispersion / solution starts to agglomerate. It is fully coagulated / flocculated at the zero point. In cases like waste water treatment, a separation of the dispersed substance from the water is intended. Flocculation of the dispersed particles and breakage of an emulsion are typical examples.
- In formulation work, the titration of an unknown dispersion or of an ionic additive to the zero point with a standard polyelectrolyte of certified charge, gives the total amount of charge. Or in other words, the effectiveness of particle coating or the electrostatic strength of additives can be determined.
- A pH titration to the isoelectric point may deliver information on the solubility of the sample (proteins).

2.3.2 Kinetics

- Coating of particles is an art. It can keep for a long time or degrade with time. The software allows measurements over long time periods with pauses responding to the behavior of the sample.
- For quality control, status measurements are programmed as short kinetics of 30 seconds or longer.

2.3.3 Critical coagulation point

In combination with size measurement, a critical coagulation point may be found in the neighborhood of an isoelectric point (metal oxides).

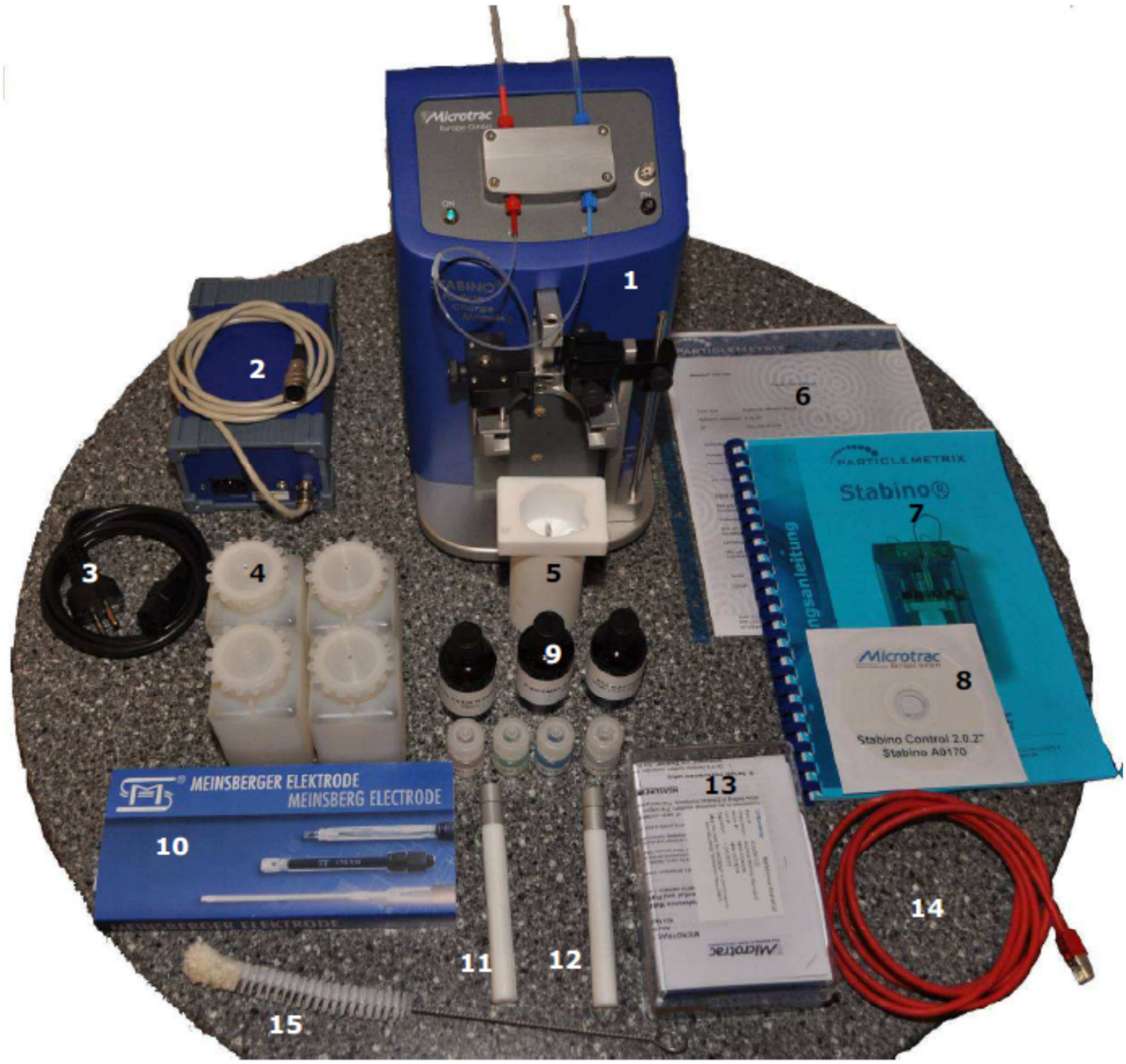
2.3.4 Agglomeration status of nano-particles

Macromolecules tend to agglomerate. The interface potential of the agglomerates is merely different from the original particles. However, the specific surface is smaller. The quantity of charge is bigger the bigger the specific surface of a particle is. In a mixture of prime particles and agglomerates, the smaller particles contribute much more to the total charge. By means of a polyelectrolyte titration the total charge can be measured. It can represent the agglomeration state of the sample. With more particles coagulated, the total charge is decreasing.

2.3.5. Specific applications

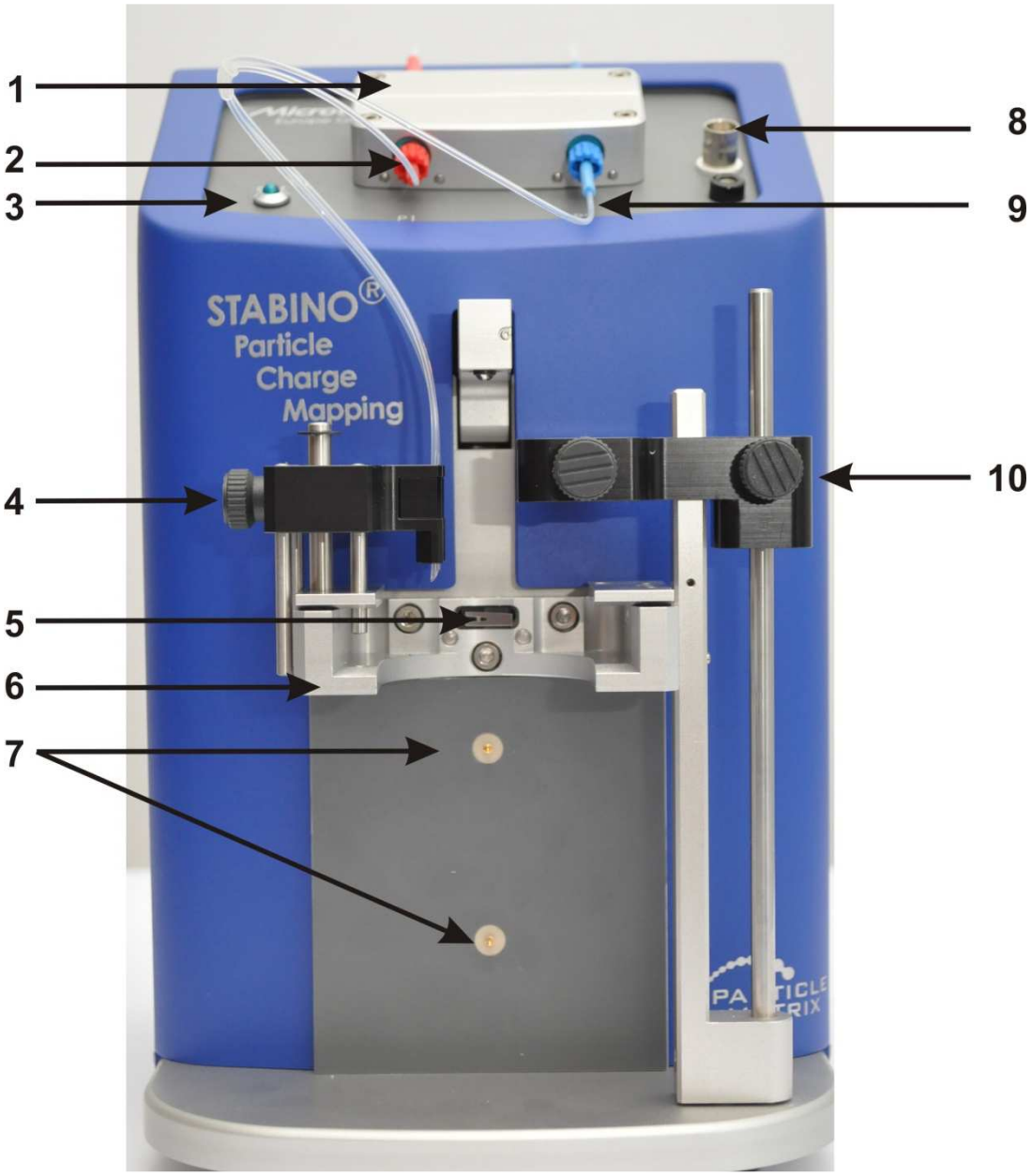
For specific applications, please read our application reports in the home page www.particle-metrix.com or in www.microtrac-europe.com.

2.4 Device description



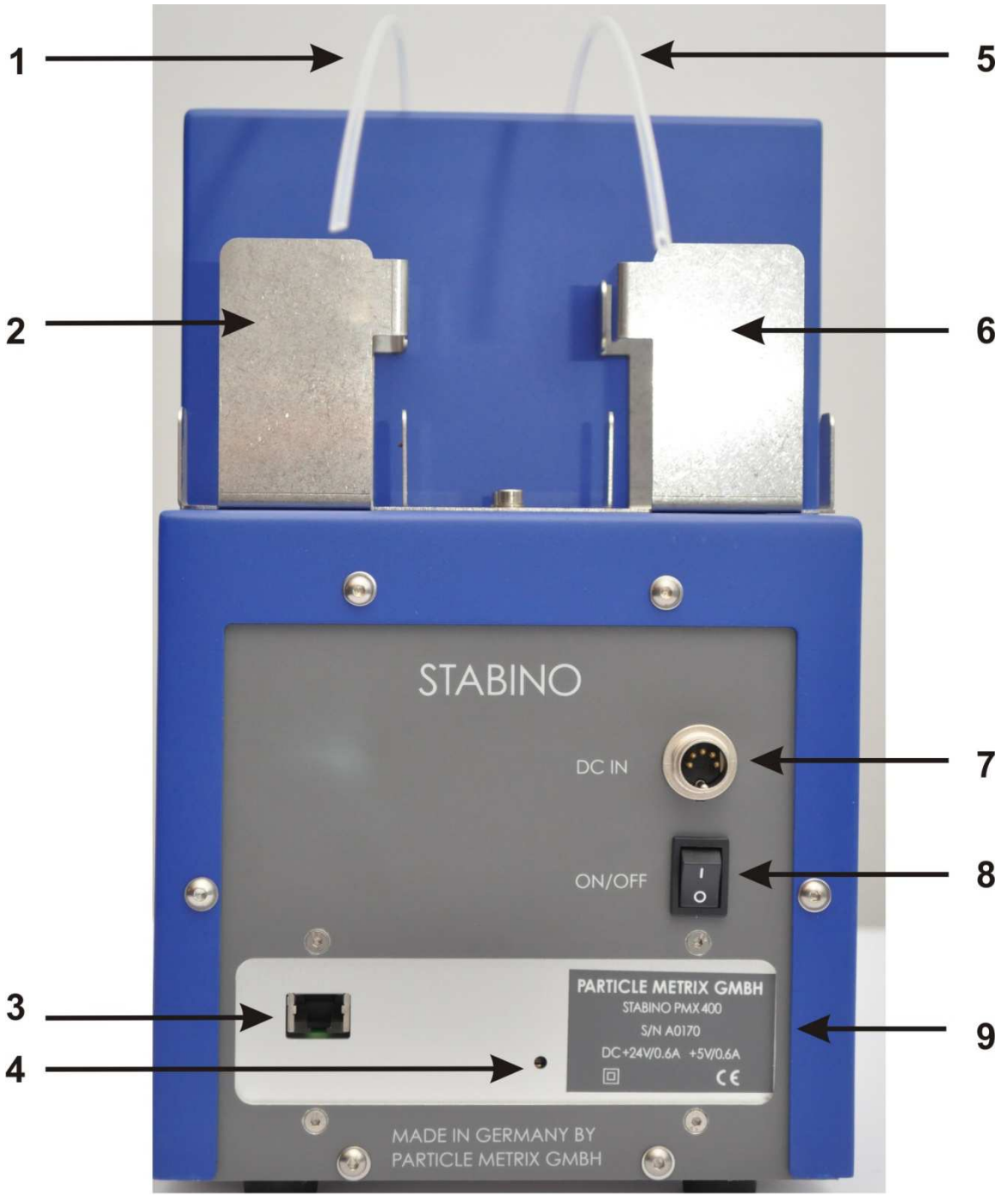
1 Stabino® including tubes and pumps
2 24 V-power supply for Stabino®
3 Power cord for the power supply of the Stabino®
4 Reservoirs for the titrant solutions (4 off)
5 Measuring beaker of Stabino®
6 Test certificate
7 Operation manual
8 Stabino®-Control Software CD including calibration data
9 Calibration solutions (P-DADMAC, PVS, KCl, and pH)
10 pH- Electrode including temperature sensor
11 Piston with 400 µm gap
12 Piston with 200 µm gap
13 Zeta potential Standard suspension + 50 mV
14 Crosslink cable
15 Cleaning brush

2.4.1 Front view



1	Housing of the pumps	6	Beaker support
2	Tubes for the titrand #1	7	Measuring electrodes
3	Power ON - LED	8	BNC – Connector for the pH Electrode and connector for temperature sensor
4	Locking mechanism of the measurement cylinder & tube holder	9	Tubes for the titrand #2
5	Detector for the presence of the measurement beaker	10	Fixing screw for pH sensor and NANO-flex probe holder

2.4.2 Back view of Stabino®



The Stabino® is powered by 24 V DC and 5 Volt DC via power source. The power source for 100-240V/50-65 Hz (optional 115 Volt) is included in the delivery package.

1 Tube for titrand #2
2 Holder for reservoir of titrand #2
3 Connection LAN-cable
4 Reset-button
5 Tube for titrand #1
6 Holder for reservoir of titrand #1
7 Power connector
8 ON/OFF switch
9 Instrument label (see 2.5)

2.5 Labeling of the Stabino®

The type plate is affixed to the rear side of the Stabino®. If this is missing, damaged or no longer legible, it must be replaced by a new plate showing identical specifications.

2.6 Technical Data

Supply voltage for the power supply	100-240 VAC 50/60 Hz
Supply voltage for Stabino	24 VDC and 5 VDC
Tolerated mains power fluctuations	+/- 10%
Fuse protection	400 mA / 800 mA (depending on country; see type plate on power supply)
Weight	8 kg Stabino® 6 kg NANO-flex (optional)
Dimensions	180 x 325 x 340 mm (W x H x D)
Protection class Stabino	II
Protection type Stabino	IP 30
Protection class Power supply	I
Protection type Power supply	IP 30
Installation	
Operating temperature	+15 to +45 °C
Air humidity in operation	< 65 % rel. humidity, non-condensing
Transport- / Storage temperature	-10 to +50 °C
Maximum sea level for operating	2000 m above sea level when using aqueous samples

Note:
When using non-aqueous dispersions, the sea level is restricted corresponding to the vapor pressure curves of the used liquids. Support from Particle Metrix is requested before using the instrument.

Data transfer

Network cable	cross-linked, CAT 5E

Sample requirements

Sample volume	1 – 10mL
Max. conductivity of sample	300 mS/cm

Dosage system

Selectable End criterion	-2000 to +2000 mV (SP) -200 to + 200 mV (ZP) pH 1 – 12
Titrant volume	0 – 20 mL manual measuring end
Resolution	10 µL

Analytical Results:

- Streaming potential
- Zeta potential
- Volume
- pH value
- Time
- Point of zero charge and isolectric point IEP
- Total charge with input of sample and titrant parameters (concentrations, weight, sample density)
- Specific surface charge with the additional input of the specific surface area of the sample from the NANO-flex size measurement at the beginning of the titration
  The viscosity is not a needed as a parameter, but may play a limiting factor for the measurement of the correct SCP when it is out of the Newtonian range. Also mixing of the titrant with the sample may be hindered.
- Conductivity
- Temperature

2.7 Scope of delivery

Packing list for Stabino® Charge titration system (view to 2.3.)

1	Basic Stabino® unit including tubes and pumps
2	24V power supply for Stabino®
3	Power cord for the power supply of Stabino®
4	Reservoirs for titration solutions (empty) (4 off)
5	Stabino® Measuring Cylinder
6	Test Certificate
7	Operation Manual
8	Stabino®-Control Software CD including calibration data
9	Calibration solutions - 0,0025 N P-DADMAC - 0,0025 N PVS, - 0,01N KCl (1,41 mS cm^{-1}) - 3 mol/L KCl - pH 4 - pH 7 - pH10
10	pH- electrode including temperature sensor
11	Piston with 400 µm gap
12	Piston with 200 µm gap
13	Zeta potential Standard dispersion + 50 mV
14	Crosslink cable
15	Cleaning brush
	Optional PC, Laptop or Tablet PC

Hint for the measurement with different pistons

The piston with a gap of 200 µm and 100 µm will be mostly used for samples with a very high conductivity over 15 mS cm^{-1} or a very low amount of particles. Measurements up to a conductivity of 300 mS cm^{-1} will be possible, but there the results of a titration will be not so good in case of the physical chemical behavior.

The piston with a gap of 1000 µm will be used for samples with a very high viscosity or for samples with a particle size over 100 µm with a maximum size of 300 µm.

Piston part-nr.	Gap / µm	Sample Viscosity	Sample Conductivity	Particle Size
200-0001 (optional)	100	low	high	< 20 µm
200-0002	200	low	medium	< 40 µm
200-0004	400	medium	medium	< 100 µm
200-0010 (optional)	1000	high	low	< 300 µm
Maximum specified		**350 mPa.s**	**300 mS/cm**	**300 µm**

Attention for titrations to 0 mV

At the zero point of charge and in its surrounding, the viscosity of the sample may drastically increase leading to a clogging in the gap. The mixing of the sample with titrant solution is not any more guaranteed. The coupling between the piston and the motor can be interrupted leading to knocking noise. The titration must be stopped and the sample cell carefully cleaned. The titration could be repeated, but at a higher sample dilution.

3. Installation

3.1 Installation requirements

3.1.1 Safety regulations

When putting the Stabino® into operation it is necessary to observe the existing safety regulations.
The device shall be exclusively put into operation and operated by appropriately trained staff.

3.1.2 Place of installation

The following points must be observed to enable a failure-free and safe use of the Stabino®:

- The ambient temperature must lie within the range of +15°C to +45°C. If the temperature of the device deviates from the ambient temperature after transport or storage, it is necessary to wait before putting the Stabino® into operation until the device temperature has adjusted to the ambient temperature.
- If condensation water has developed during transport or storage, it is necessary to wait 2 hours for acclimatisation.
- The device has to be installed on a stable, flat worktop.
- The device must be operated in a dry and clean environment.
- Avoid direct sunlight on the device.
- The device must not be operated in explosion prone rooms.
- Protect the device against dripping and splashing water.
- The device may only be operated when connected to ground type socket with a mains voltage of 100 – 240 V, 45 – 65 Hz.

3.2 Installation of Stabino®

All following advices refer to the screen shots in chapter 2.4.

Security note

Check, whether the Stabino is switched off („ON/OFF"-switch on the rear panel to position „O"), before you plug the power supply into the mains socket. Wait with switching ON the instrument until this is described in chapters 3.2.1 bzw. 3.2.2.
Use a mains socket nearby the instrument for rapid unplugging, if needed.

Socket on the rear for the DC power supply (24 Volt)

Main ON/OFF switch

3.2.1 Connecting Stabino® via LAN

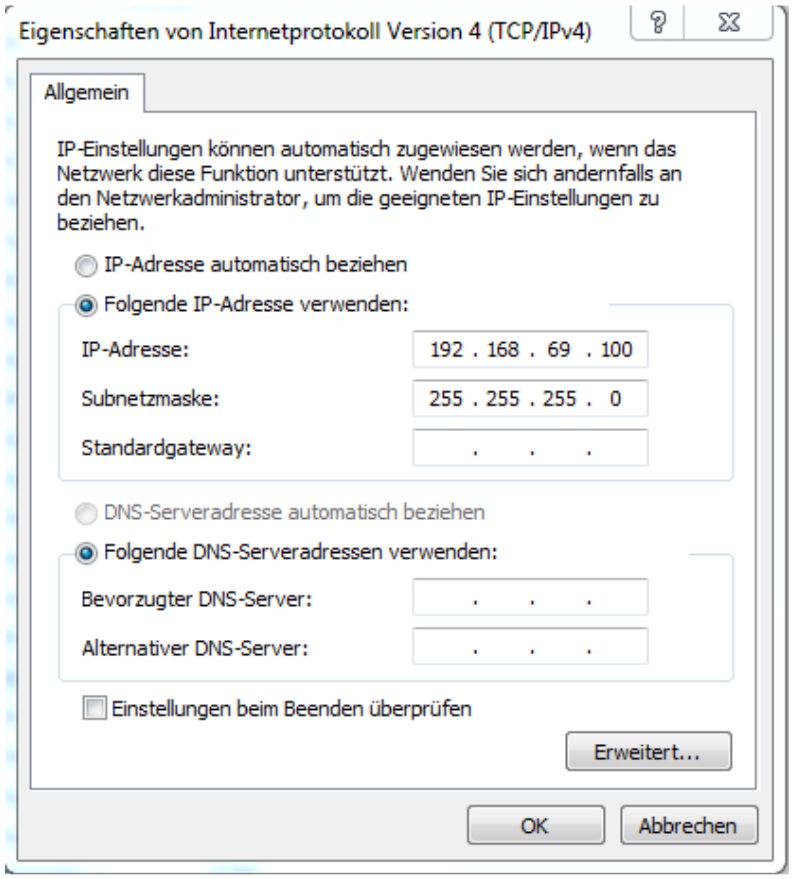
- Connect Stabino® with LAN cable (twisted-pair cable) with the PC
- Connect the power line with the Stabino®
- Put the power cable into the socket
- Turn on Stabino®
- Turn on the PC (follow the instruction manual of the PC)

3.2.2 Setting up Stabino® on the PC

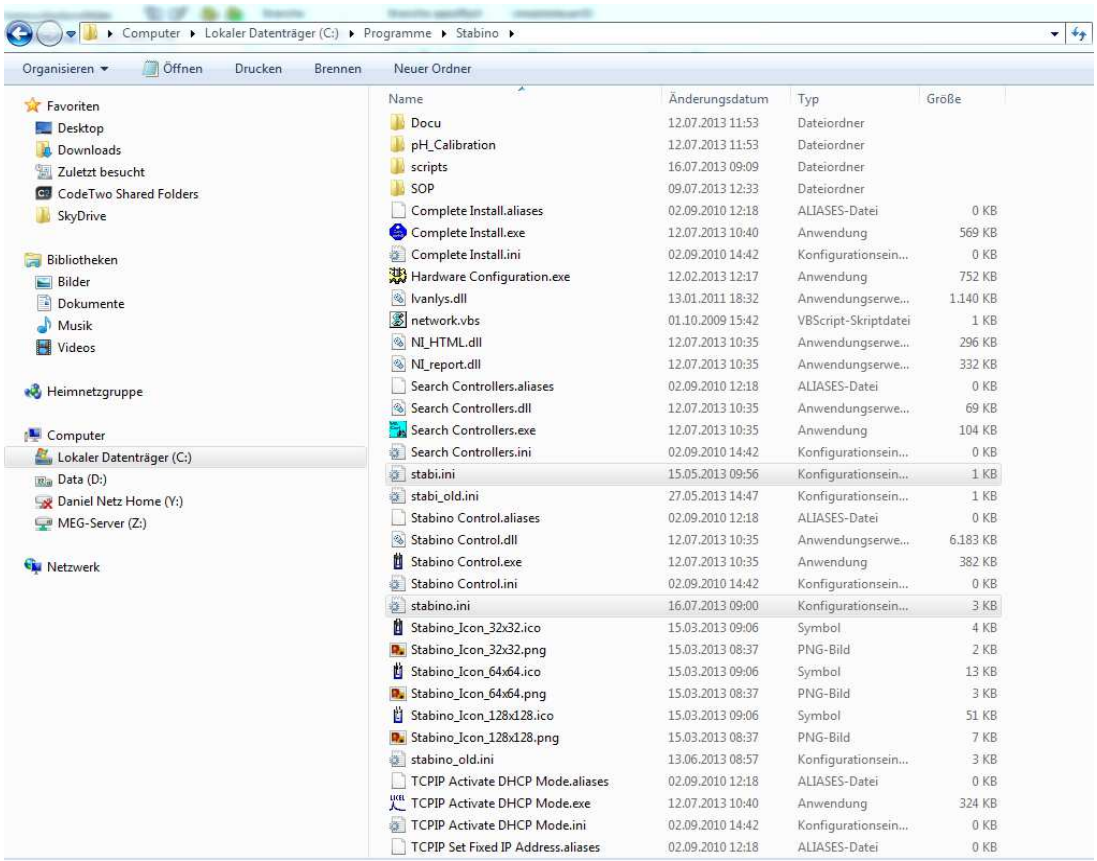
- Install the Stabino Software to the PC

- Thr Stabino is set to a fixed IP-Address. The standard address room is 192.168.69.xxx.
  xxx stands for the last 3 digits of the serial number
  (After a hardware reset the IP changes automatically to 10.49.234.234)

- Set the IP address of the PC (fix) to 192.168.69.100 Sub.Net. 255.255.255.0



- At the end, please copy the files "stabi.ini" and "stabino.ini" into the Stabino Control folder. The calibration data are stored on the software CD.



3.2.3. Starting the Stabino®-Software

The desktop shows two program icons, "Microtrac-FLEX" for 180° DLS size measurements with NANO-flex and "Stabino Control" for the particle charge potential and titration measurement with Stabino®. If both instruments are connected, both instruments can be simultaneously operated.

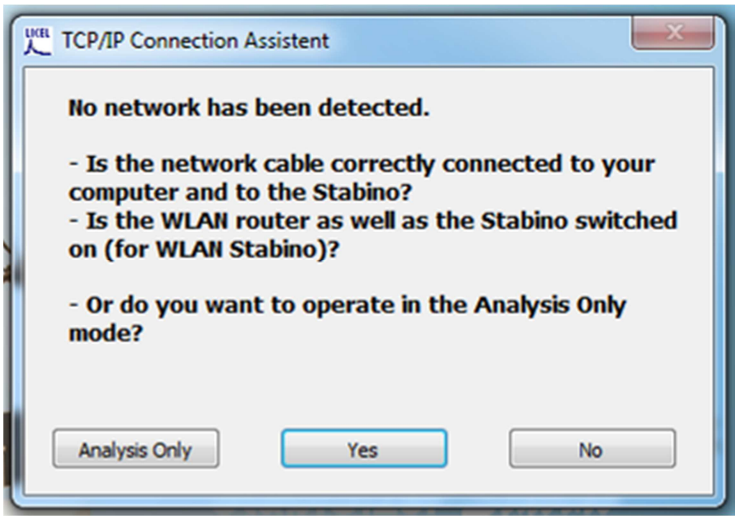
- Start the **particle potential and titration software** with „Stabino Control".
- The registering window opens to enter user name and password. At delivery, no user name and no password are defined. By clicking "OK" the program starts without entering a user name and a password. The home menu appears.

Please note: User name and password can only be given in the "admin"-mode. This refers to software versions later than 2.00.16.

- In case you forgot your password, please contact your administrator.

After successful registration the „home" page of Stabino® appears.

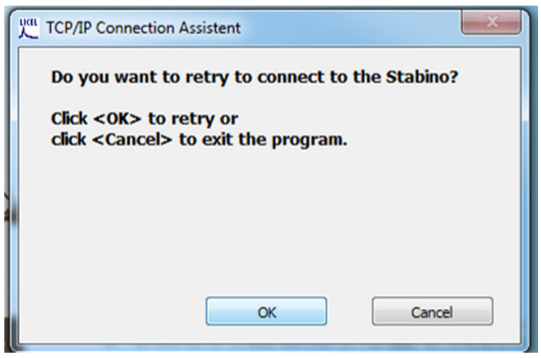
If the software was started without having connected the PC with the Stabino® following message appears:



You have following choices to answer:

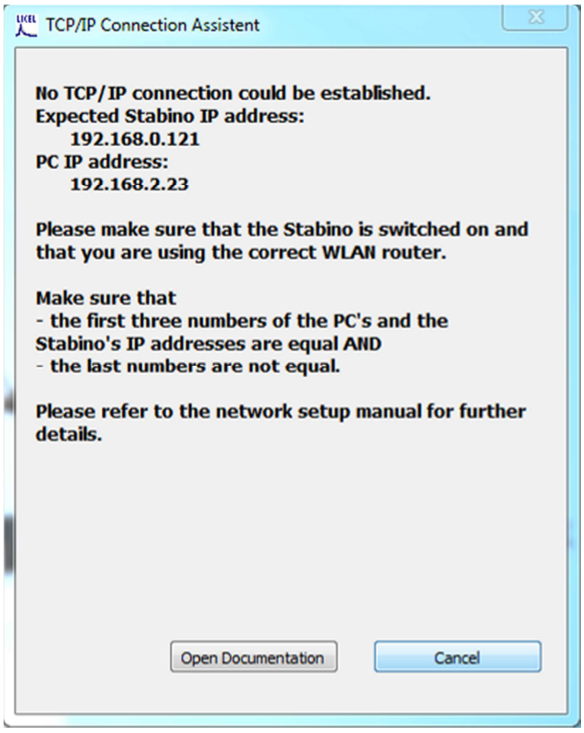
Analysis Only: You arrive in the analysis mode without the need to have a connection of the PC to the instrument. In this mode only the functions "Analysis", "SOP", "Global Settings", "Service" and "Help" can be selected.

Yes: The software tries to get a network connection to the Stabino®. Following window opens:



By pressing "OK", the system tries to establish a new connection and the message appears:

Please wait a minute. When connected you are in the log-in mode.
If no connection establishes, the window hereafter opens. Please press "Open Documentation" and follow the instructions of this document.



"Cancel" closes the Stabino® Software.

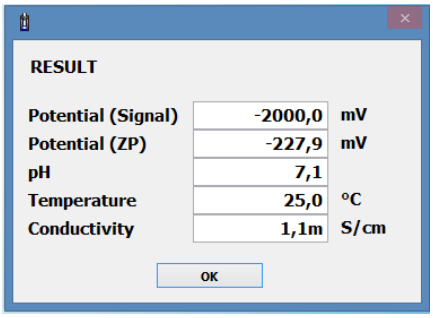
Advice
By pressing "**STRG + H**" you will get a short help message ("Kontexthilfe").

4. Measurement

4.1 Sample requirements

Many samples are measurable with Stabino® without any specific preparation. The quickest way to test the measurability is outlined here. The measurement is started with the sample as it is. Is the measured potential stable and if the system does not produce unknown noises from the measurement cell, the sample measurement can be started. To test the sample please select "**Measurement"** / "**Check Sample"**.

The actual potential, the pH-value and the temperature are displayed in a window. In addition, the polarity of the charge cationic (+) or anionic (-) is given. If the pH-sensor is not connected, no pH reading appears. These data are not memorized. They have informative character only. If a titration should follow, the data indicates, in which direction a titration should be programmed.



Advice
The consumption of titration solution should be between 1.5 and 9 mL. If the consumption is outside of that range, appropriate dilution of the titrant solution and the sample respectively, brings the consumption into the allowed region.

In the following a few recommendations for the sample preparation are presented.

4.1.1 Utilization of solvents

The measurement of the potential is only possible in polar media and water. In general, charge can be analyzed by dissociation of functional groups. Samples which are not solvable in water may be dissolved in lower alcohols and diluted thereafter in deionized water. The optimum mixing proportion has to be evaluated by experiments to get accurate measurement results, it is recommended to perform a reference measurement with the selected alcohol water mixture. The hereby obtained result should be deducted from the following measurements with sample.

Attention
When using solvents, the corresponding rules for hazardous products have to be followed.

Attention
pH- measurements and titrations are allowed in aqueous media only. Organic media cause damage of the pH electrode.

4.1.2 Practicable particle size range

The mechanical design of the measurement cylinder and the piston limits the maximum possible particle size to 100 µm. As an exemption, with a special piston the upper limit can be extended to 300 µm. **The lowest measurable particle size is 0.3 nm.**

.

The mechanical design of measurement cylinder and piston is such that the maximum particle diameter is limited to 1/3rd of the piston gap.

100 µm piston:	maximum particle size	< 30 µm
200 µm piston:	maximum particle size	< 60 µm
400 µm piston:	maximum particle size	< 100 µm
1000 µm piston:	maximum particle size	< 300 µm

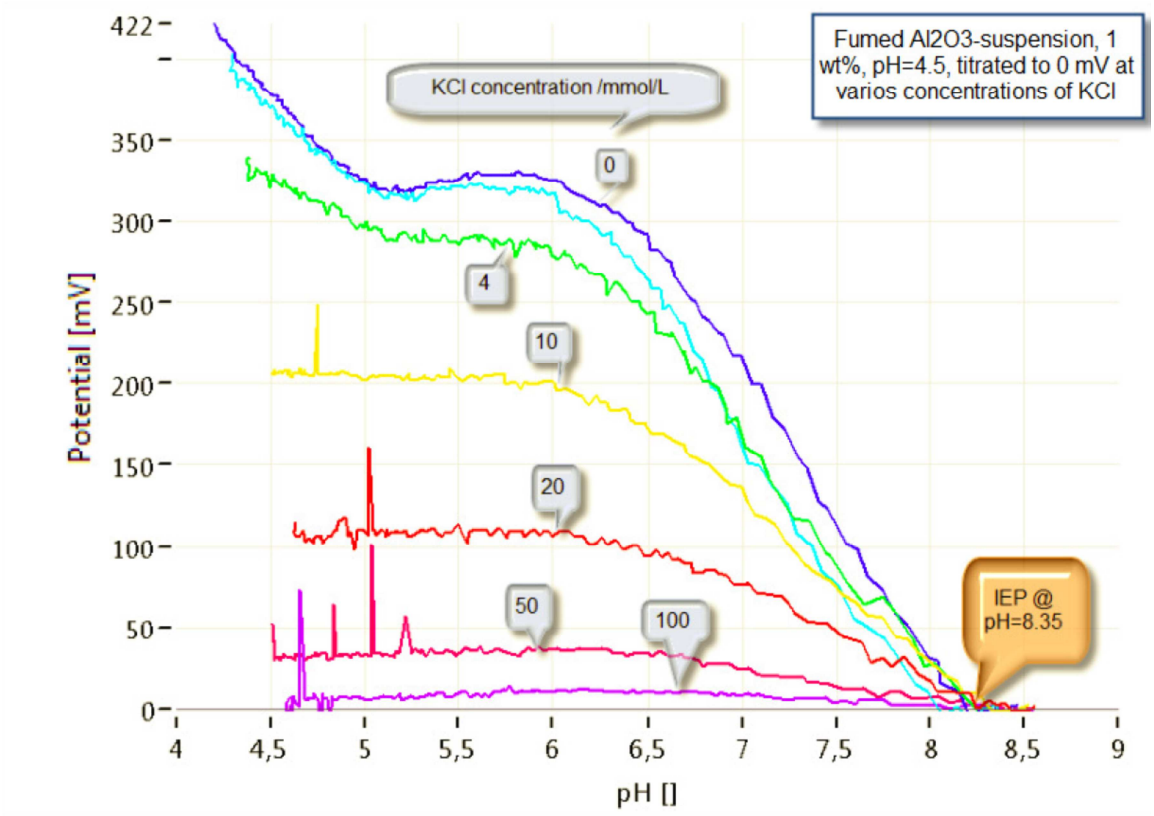
4.1.3 Viscosity

With the standard measurement cell assembly samples with a viscosity up to 300 mPa*s can be measured. Samples up to 5 Pa*s can be measured with the 1.000 µm – gap piston. Samples with too high viscosity should be diluted.
A too high viscosity is indicated acoustically by noises from the cell or from the coupling magnet, which is situated on top of the piston. Additionally, a warning from the software may appear. "Slip…"

4.1.4 Conductivity

The measured potential is strongly dependent from the conductivity. With rising conductivity, the solution approaches a competitive short circuit situation. As a consequence, the potential decreases.

The conductivity is constantly monitored and documented by Stabino®. In the following graph, the influence of the conductivity on the measurement signal is documented.



With increasing conductivity the potential decreases. This is a consequence of the decreasing double layer thickness.

Advice

Please note, that the character of the particle interface expressed as the isoelectric point remains unchanged.

4.2 Sample preparation

The measurement result is strongly dependent from the way the sample is prepared and how it is presented to the measurement cell. Therefore it is important to prepare the sample carefully and invariably in the same way to guarantee comparable good results.
On the other hand, this dependence of the results from sample preparation can be exploited by purpose to get more information about the behavior of the sample in relation to the charge environment.

4.2.1 Centrifugation of coarse particles

To get rid of coarse particles it is recommended to keep the same centrifugation conditions constant. In particular the use of the same centrifugal force and the same duration of centrifugation have to be applied.

4.2.2 Sedimentation to separate coarse particles

If the sample contains solid particles with tendency for rapid formation of a sediment, the supernatant can be taken with a pipette and analyzed. For reproducible results it is important to keep the duration of sedimentation constant.

Notice
By centrifugation and sedimentation it is possible to determine the charge of the particles and the charge of the colloidal solution individually.

4.2.3 Filtration to separate coarse particles

During filtration through a filter cake or a paper filter polyelectrolytes may be adsorbed. Consequently, the reproducibility of results could suffer from this adsorption. It is therefore recommended to refrain from filtering samples.

4.2.4 Sieving to separate coarse particles

Sieving is a simple and fast method. The elimination of big particles and fibres from the sample is quite sufficient for a measurement.

4.3 Preparing Stabino® for the measurement

Attention
For the cleaning of the measurement cylinder and the piston it is advised to use the delivered brush and the cleaning solution described in chapter 6.1.

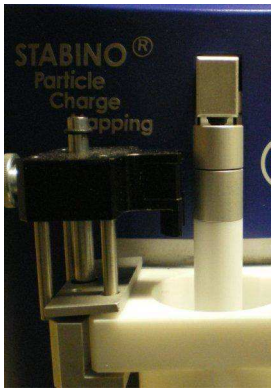
Attention: The electrical contacts at the rear of the measurement cylinder must be kept dry and clean.

- Depending on the sample cylinder volume, please fill 10 mL or 1 mL of sample into the clean sample cylinder.
- Please make sure that the locking mechanism of the cell is in the upper position.
- Slide the carefully cleaned piston into the measurement cylinder. The sample must not spill out during this action.
- Apply the optional spill protection.

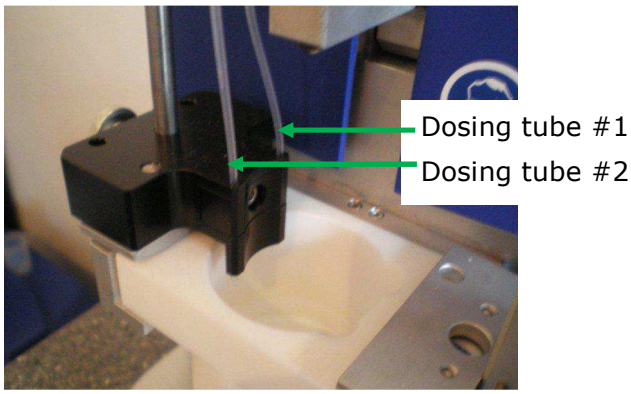



- Slide the measurement cylinder into the sliding rail all the way to the stop. The contacts must point to the instrument. The piston will be automatically pulled up by the magnet and brought into the measurement position.

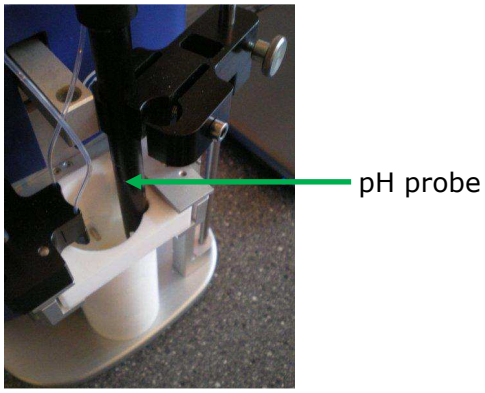
- Fix the position of the measuring cylinder by pulling down the locking mechanism on the left side.



- Slide the dosing tube through the holder right on top of the measurement cell, so that the titrant solution is free to drop into the measurement cylinder.



- Mount the pH probe into its holder situated in the back position. The sensor fits exactly into notch of the Teflon beaker.



Notice
Thorough cleaning of the measurement cell and the exact dosing of the probe affect the reproducibility of the measurement results.

Notice
If the 180° DLS size sensor is installed, you first have to remove the size sensor, before you remove the measurement cell. After lifting the locking mechanism, the

measurement cell together with the piston may be pulled out towards the user. It is not necessary to manually decouple the piston from the magnet!

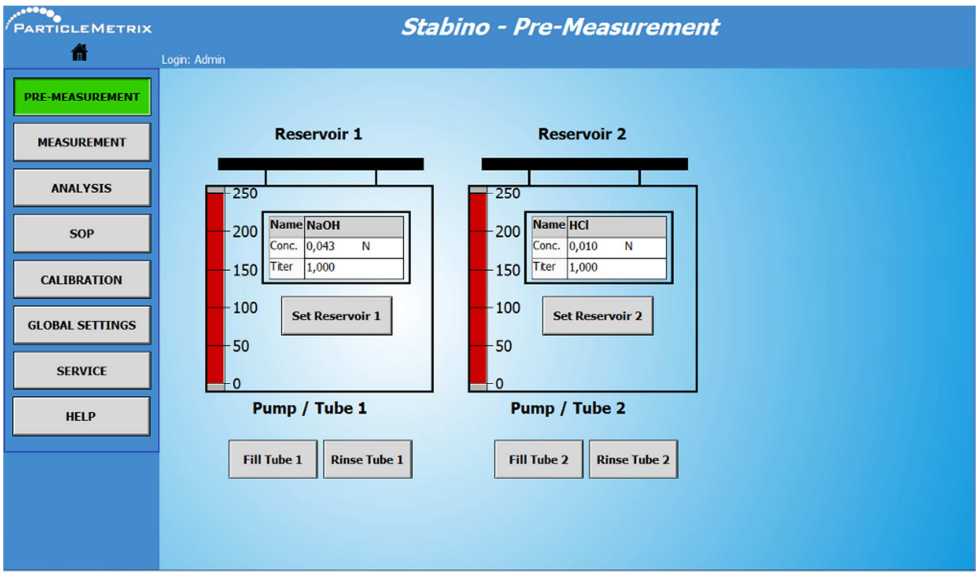
4.3.1 Preparing a titration

If the instrument is new, please perform first the calibrations as described in chapter 7 – "Calibration of the Stabino®".

After the Stabino® is calibrated go to the menu "**Pre-Measurement**".

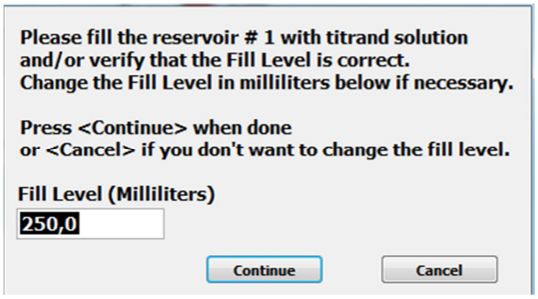
The name of the titration solutions can be entered in the boxes "Name". The concentration of the titrant solution is entered into the box "Conc." If a titer is available it is entered into the corresponding box "Titer". Is no titer determined, the value in the field remains = 1. **The values in the boxes "Titer" and "Conc" are used for further calculations.**

Attention!
Wrong entries in the boxes "Titer" and "Conc." may give rise to wrong measurement results.



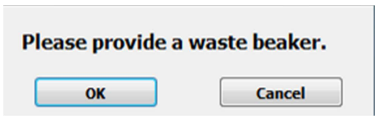
Via the function 

you can enter the volume of the titrant solutions in the containers ("reservoirs").

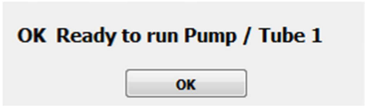


Notice
If the value of the volume is not carefully entered, the measurement may be interrupted. The volume calculates the remains in the titrant bottles and if the volume decreases below the value of 5 mL, the titration is stopped and a warning message appears with a prompt to replenish the titrant solution. After refill, the titration can be continued.

- In order to fill the pumping system and the tubes, press "Fill Tube". A prompt appears to "provide a waste beaker" under the exit tube. With "OK" you continue. In the case the measurement cell is in place, a window appears asking to remove it. After confirmation, a new window asks to "provide a waste beaker". Only by then the filling procedure for the pump and hose system is possible. The fill procedure is blocked as long as the measurement beaker is not removed.



- During the filling procedure it is recommended to check whether bubbles appear in the hoses. Gentle tapping onto the hoses helps to chase the bubbles.



- Now, the filling procedure for the pump #1 system is finished. To fill pump system #2 proceed as described for pump #1.

Repeat the accomplished steps, if bubbles happen to stay in the hoses. It is rudimentary for a good titration, that the hoses remain bubble free.

Note
Provided you want to switch to another titration solution or to change to a solution of different concentration or to a solution of opposite character (anionic to cationic polyelectrolyte or vice versa, base to acid or vice versa) it is necessary to rinse the dosing system of the Stabino® via the „Rinse Tube" function in "Pre-Measurement". In this way interference or a reaction (flocculation) in the dosing system can be avoided.

4.4. Creating operating methods (SOPs)

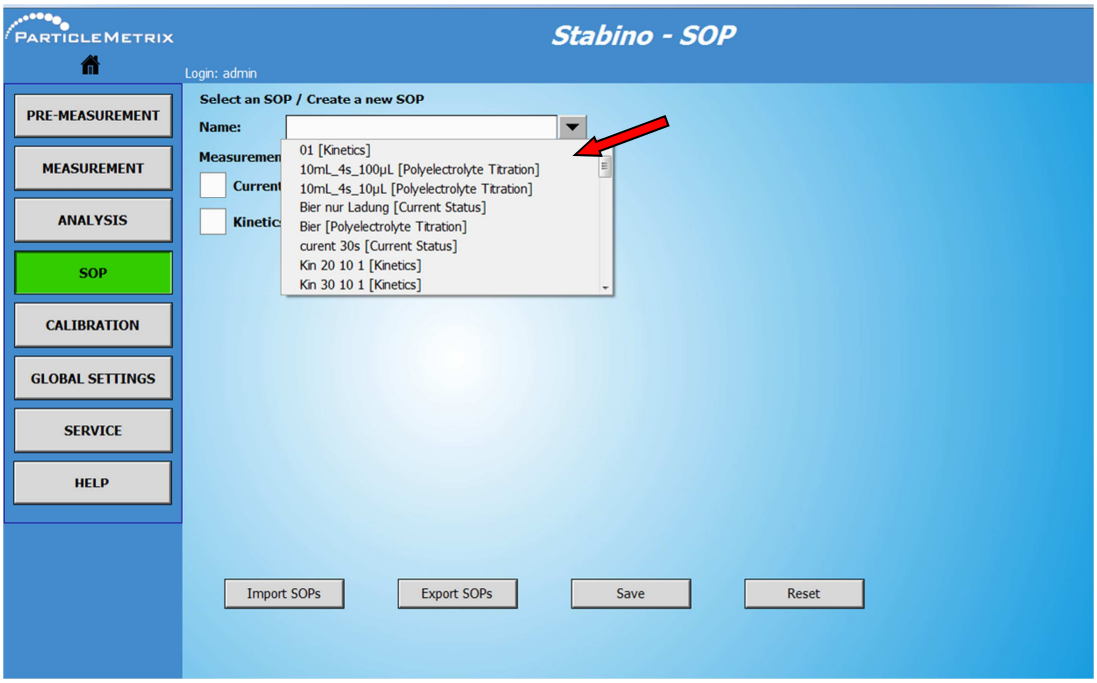
This chapter describes how to set up **S**tandard **O**perating **P**rocedures (SOP) type "SOP".

In the opening window you select the desired SOP.

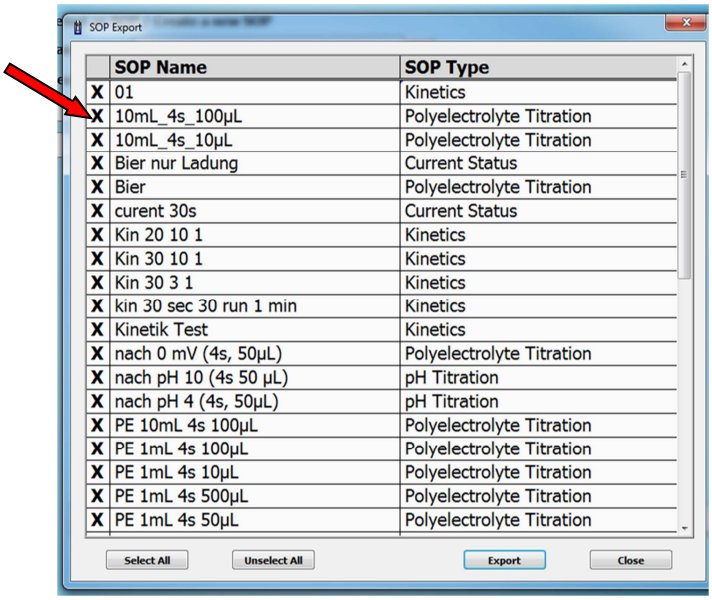
Attention!
Before you can perform a measurement, the adequate SOP must be already defined.

Following measurement methods are available:

Measurement type	description	Value for Y-axis	Value for X-axis
Current Status	Simple measurementof the potential	• potential	• time
Kinetics	Meassurement of the potential over a longer time with measurement interrupts between single measurements	• potential	• time
pH Titration	Titration to the isoelectric point IEP (0 mV) or to a special pH value	• potential	• pH
Polyelectrolyte Titration	Titration to the point of zero charge or monitoring the potential over an extended range of polyelectrolyte	• potential	• volume • molar quantity • Coulomb
Salt Titration	Titration to monitor the potential over an extended range of electric charge	• potential	• volume • Coulomb

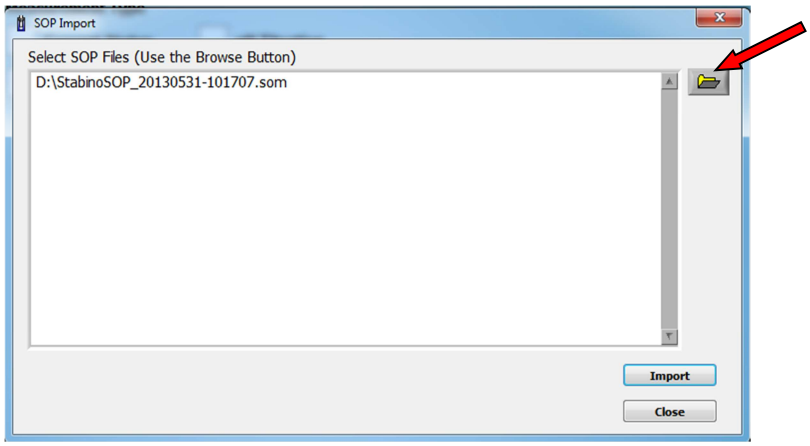


By activating the dropdown menu (red arrow) the window opens, where all SOP sappear. Beside each SOP the type of measurement is indicated in brackets. The SOP opens by clicking on to it. The settings are visible and can be modified. With the export button they can be exported. Following window opens by click.

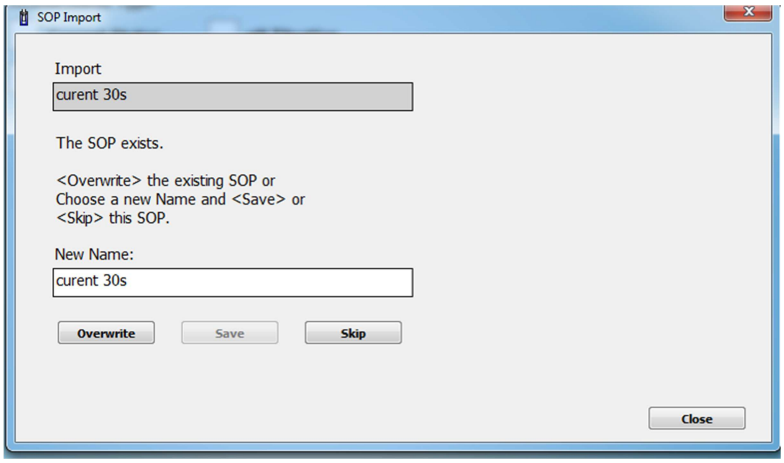


In this list of SOPs all SOPs can be selected or unselected. Single SOPs chosen to be exported may be selected by tipping on one of the SOPs in the first column (red arrow). Click “Export” and select the folder into which the file should be exported. The files are exported with the extension *.som.

For the import of SOPs click “Import SOPs” and select the location where the *.som file is stored (red arrow). After seletcing a file, it is displayed in the white window. Now klick on button “Import” to import the selected SOP.



If an SOP of the same name exists already, following window opens:



Here you can choose, whether you would like to overwright the present SOP or to give a new name to it or to skip the SOP.
After successful import of the SOP klick „Close". Herewith, the import procedure is finished.

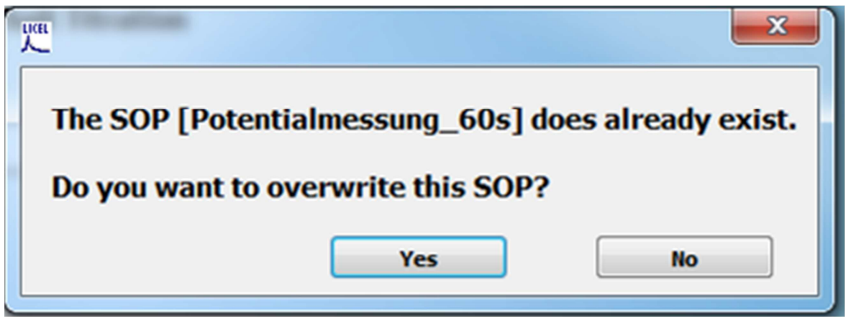
The different types of SOPs are explained in the following chapter.

4.4.1 Current Status

The method "Current Status" is a "simple" measurement of the potential without titration. The end point of the measurement is just the entered time. Please enter the name of the SOP into the input field "Name" and save it by clicking "Save".

Notice
It is recommended to designate the SOP with a name, from which it is obvious which measurement settings were chosen. Example: „Potentialmessung_60s"

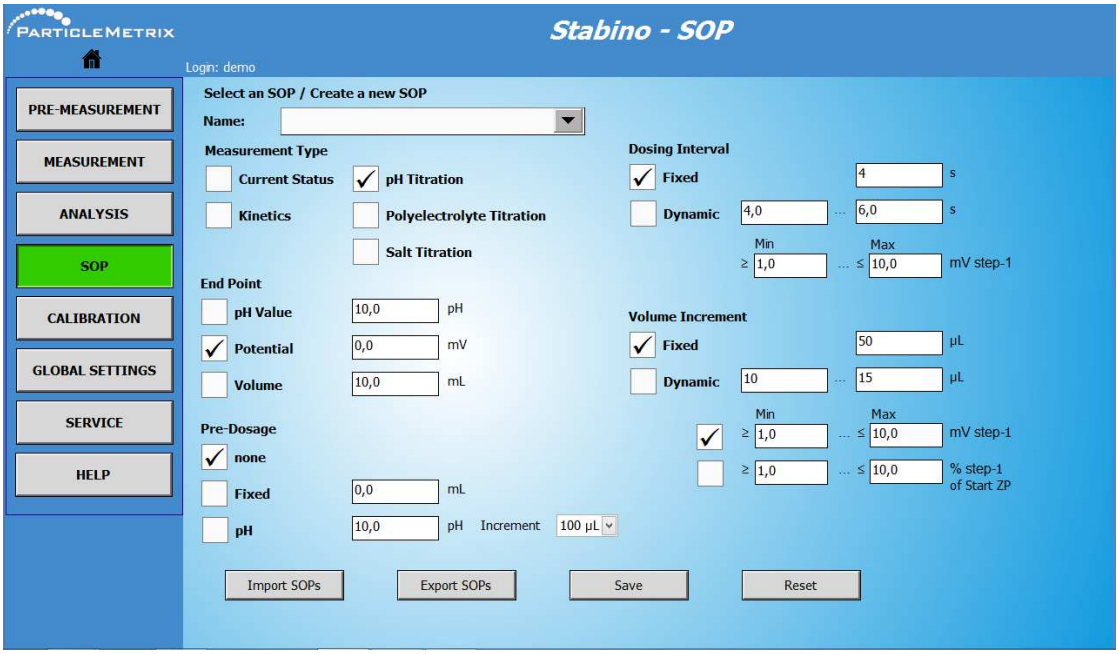
Attention!
If the SOP name is already assigned, a notice window appears that the name already exists. You may overwrite the SOP or to find a new name.



4.4.2 Kinetics

The measurement method "Kinetics" is a "simple" measurement of the potential without any titration. With this measurement type several "current status" measurements are made in series. Measurement duration, number of measurements and waiting time between the measurements are defined by the user. Please enter the name of the SOP and confirm it with "Save". Please read the notice under chapter 4.4.1.

4.4.3 pH titration



Most of the time, the IEP iso-electric point is determined with the measurement method "pH Titration". But it is also possible to perform a "screening" of the sample over a certain range of pH.

The end point of a titration can be defined as follows:

1. pH-value: A certain pH value can be chosen as end point. Acid or base solution respectively, are added as long as the chosen end point is attained.

2. Potential: A fixed potential value can be chosen as the end point. Example: Potential = 0 mV (IEP). Acid or base, anionic or cationic polyelectrolyte solution, respectively are added as long as the chosen end point is attained.

3. Volumen: A further posibility is to titrate as long as a certain volume of titrant solution (acid/base, cationic or anionic polyeleoctrolyte) is added.

Attention!
A maximum of 20 mL of titrant solution can be added to a sample volume of 10 mL. After 20 mL the titration is automatically stopped to avoid overflow of the measurement cell. Therefore the **maximum sample volume must be restricted to 10 mL**.

Further settings:

Pre-dosage: The entered volume will be pumped into the sample, before the proper titration measurement starts. The pre-dosage setting abbreviates the experiment with familiar samples.

Dosing interval: **Fixed:** The dosing pumps pump at a fixed frequency. The waiting time between titration steps can be set between 4 up to 100 sec.

Dynamic: A flexible waiting time between titration steps can be set. Within this time interval the fluctuation of the signal is expected to achieve a value below the set limit in mV/sec. The waiting interval can be chosen between 3 and 100 sec. The fixed waiting interval is inactive. Minimum interval, maximum interval time and signal are active.

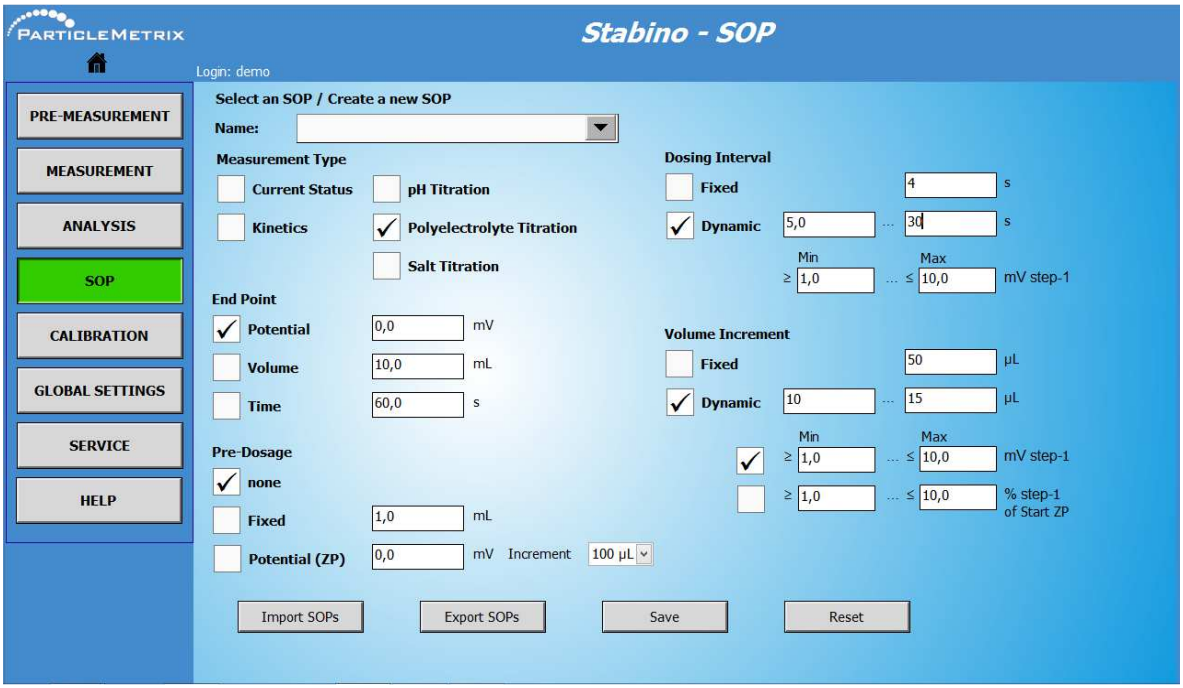
Volume increment: **Fixed:** A fixed volume is dispensed.

Dynamic: A maximum volume to dispense is set. The software calculates the necessary volume to keep the signal within the allowed fluctuation of the signal.

Attention!
With dynamic titrations, the end points are only defined by pH and potential. The sampling intervals are defined in the following way:
- pH titration: ΔpH / step
- Potential titration: Δς / step

Please enter the name of the SOP into the box “Name” and save it by clicking “Save”. Please read also the notice under chapter 4.4.1.

4.4.4 Polyelectrolyte Titration



With a **polyelectrolyte titration** it is possible to determine the point of zero charge (@ 0 mV) and deducted from the volume consumption to the zero point of charge.

The end point of the polyelectrolyte titration can be defined as follows:

1. Potential: You have the choice to set a fixed potential value as end point, where the titration automatically stops. Most frequent: Potential = 0 mV (Pointo of zero charge). Cationic or anionic polyelectrolyte solutions are added, until the potential = 0 mV is achieved.

2. Volume: You have the choice to define a volume of polyelectrolyte solution, which should be dispersed into the sample.

3. Time: You have the choice to determine a duration, within what time polyelectrolyte solution should be added.

Attention!
Maximal dispense of 20 mL is possible to add to a sample of 10 mL volume. After addition of 20 mL volume, the titration is automatically stopped to avoid overflow of the measurement cell. Therefore the **maximum sample volume** should be limited to **10 mL**.

Further settings:

Pre-dosage: The entered volume will be pumped into the sample , before the proper titration measurement starts. The pre-dosage setting shortens the experiment with familiar samples.

Dosing interval: **Fixed:** The dosing pumps pump at a fixed frequency. The waiting time between titration steps can be set between 4 up to 100 sec.

Dynamic: A flexible waiting time between titration steps can be set. Within this time interval the fluctuation of the signal is expected to achieve a value below the set limit in mV/sec. The dispensed volume is then increased or decreased accordingly. The waiting interval can be chosen between 3 and 100 sec. The fixed waiting interval is inactive. Minimum interval, maximum interval time and signal are active.

Volume increment: **Fixed:** A fixed volume is dispensed.

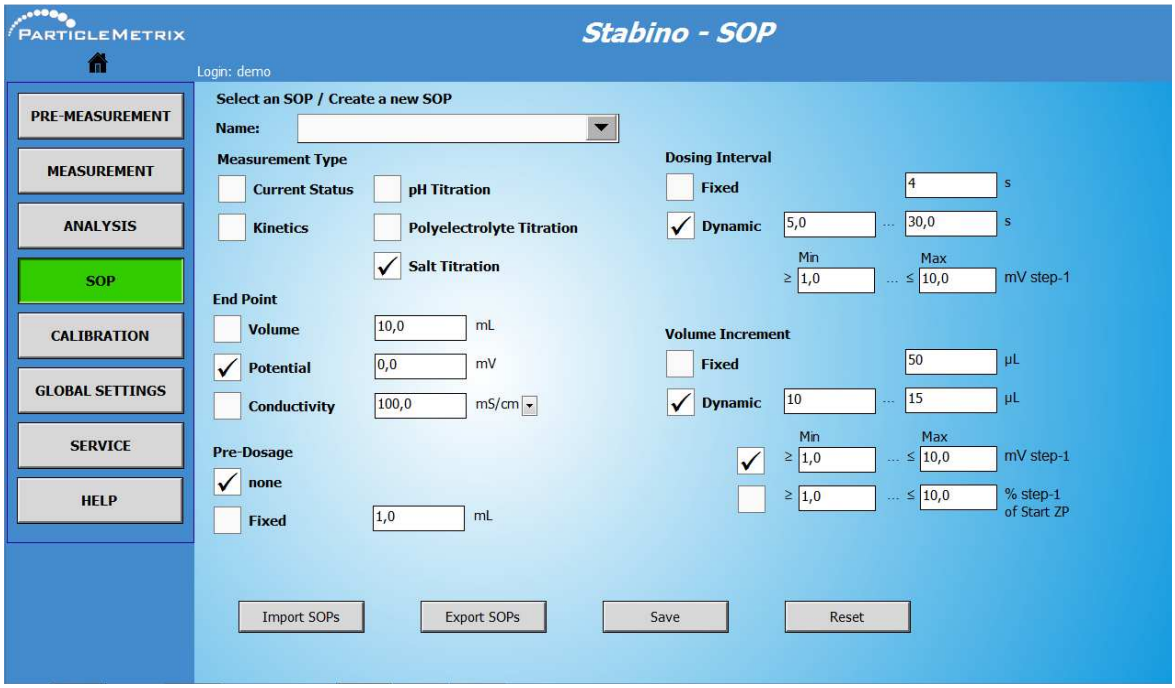
Dynamic: A maximum to dispense volume is set. The software calculates the necessary volume to keep the signal within the allowed fluctuation of the signal.

Attention!
The dynamic titration is only possible with an end point entered in "mV". The maximum titration steps are defined in "ΔmV/step".

Please enter the name of the SOP into the box "Name" and save it by clicking "Save". Please read also the notice under **chapter 4.4.1.**

4.4.5 Salt Titration

With the measurement method "Salt Titration" the influence of the electrolytic environment of the sample can be measured.



The end point of the titration can be defined as follows:

1. Volume: You have the choice to define a volume of salt solution, which should be dispersed into the sample.

2. Potential: You have the choice to set a fixed potential value as end point, where the titration automatically stops. Most frequent: Potential = 0 mV. The salt solution is added, until the selected potential is achieved.

3. Conductivity: The end point of the conductivity titration is defined in conductivity.

Attention!
Maximal disposal of 20 mL is possible to add to a sample of 10 mL volume. After addition of 20 mL volume, the titration is automatically stopped to avoid overflow of the measurement cell. Therefore the maximum sample volume should be limited to 10 mL.

Informations about further settings can be gathered from Chapter4.4.4.

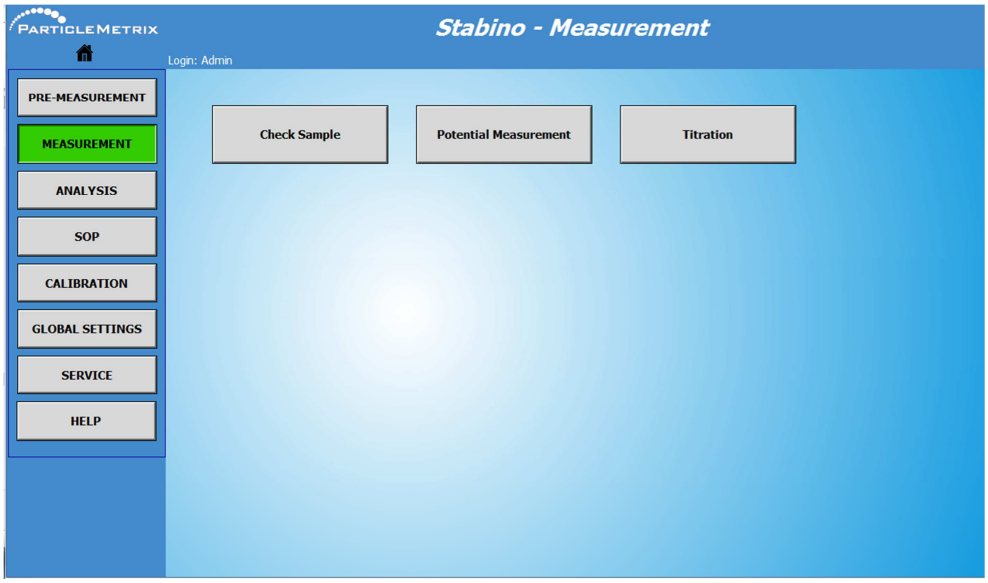
Please enter the name of the SOP into the box "Name" and save it by clicking"Save". Please read also the notice under chapter 4.4.1.

GENERAL COMMENT ON TITRATIONS

With titrations it is possible to find the isoelectric point IEP of a dispersion, where the dispersion becomes totally unstable. From the shape of the titration, stable zones can be identified. The potential of the stable zones are far away from zero potential. It is recommended to prove stability with a 180° DLS experiment with the optional NANO-flex instrument. If the size distribution is mono-modal and does not show agglomerate peaks, the dispersion is likely to be stable. See chapter 4.5, subtitle "size measurement".

4.5 Measurements with Stabino®

In order to initiate a measurement switch to the menu "Measurement". You have the choice between three main groups of measurement types.

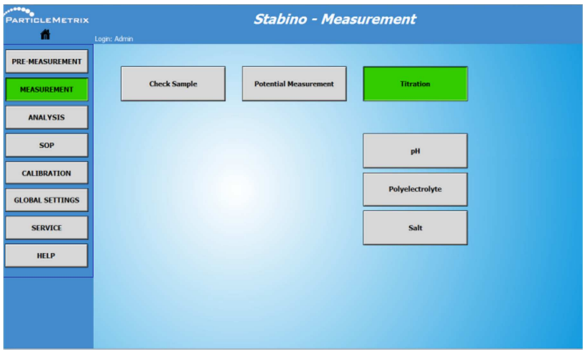


Check Sample: A short check of the sample is performed with "Check" as described in chapter 4.1.
Please note that the data are **not saved!**

Potential Measurement: Under this measurement type all measurements are pooled which do not involve titrations (Current Status und Kinetics).

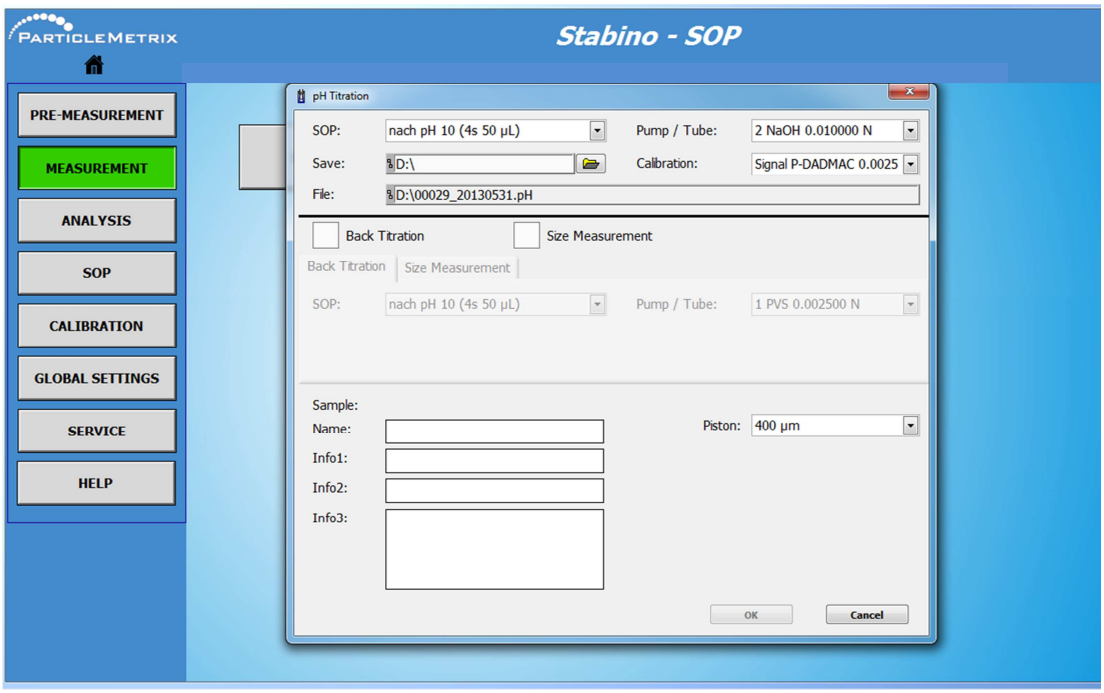
Titration: Under this measurement type all titration measurements are pooled (pH, Polyelectrolyte und Salt).

As soon as you select a pool menu, the measurement types in this group can be seen.



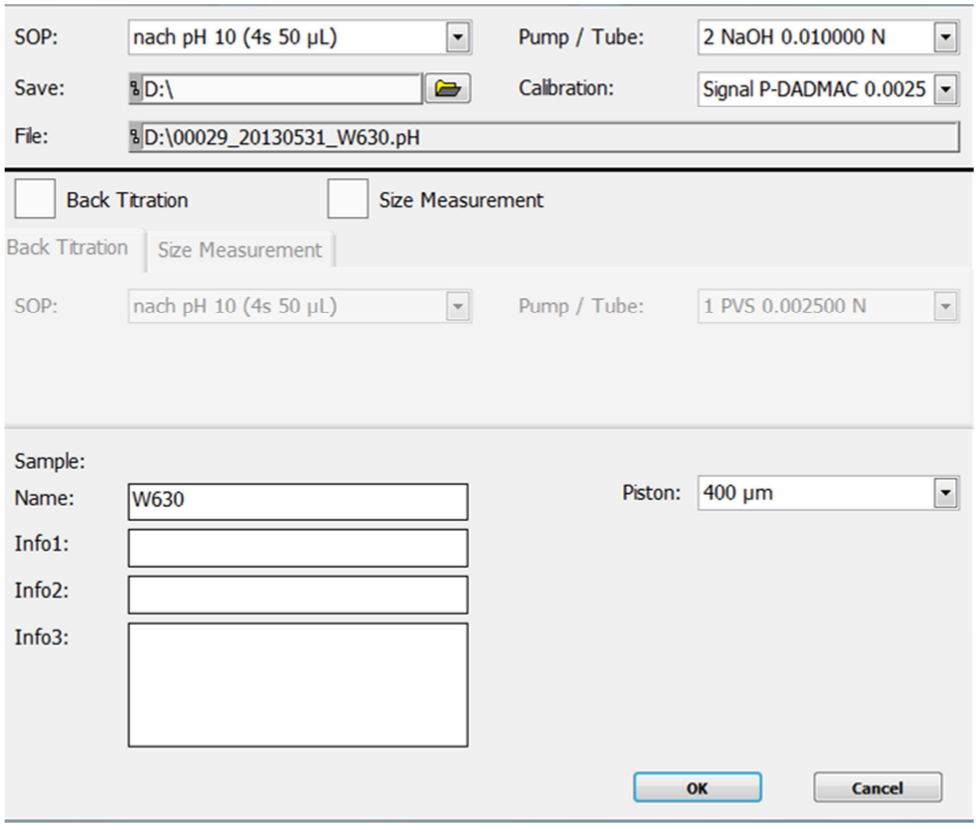
To start a measurement, click on the desired sub-menu. In the following the initiation of a measurement is shown in the example of a pH titration.

By clicking on pH a dialogue window opens:



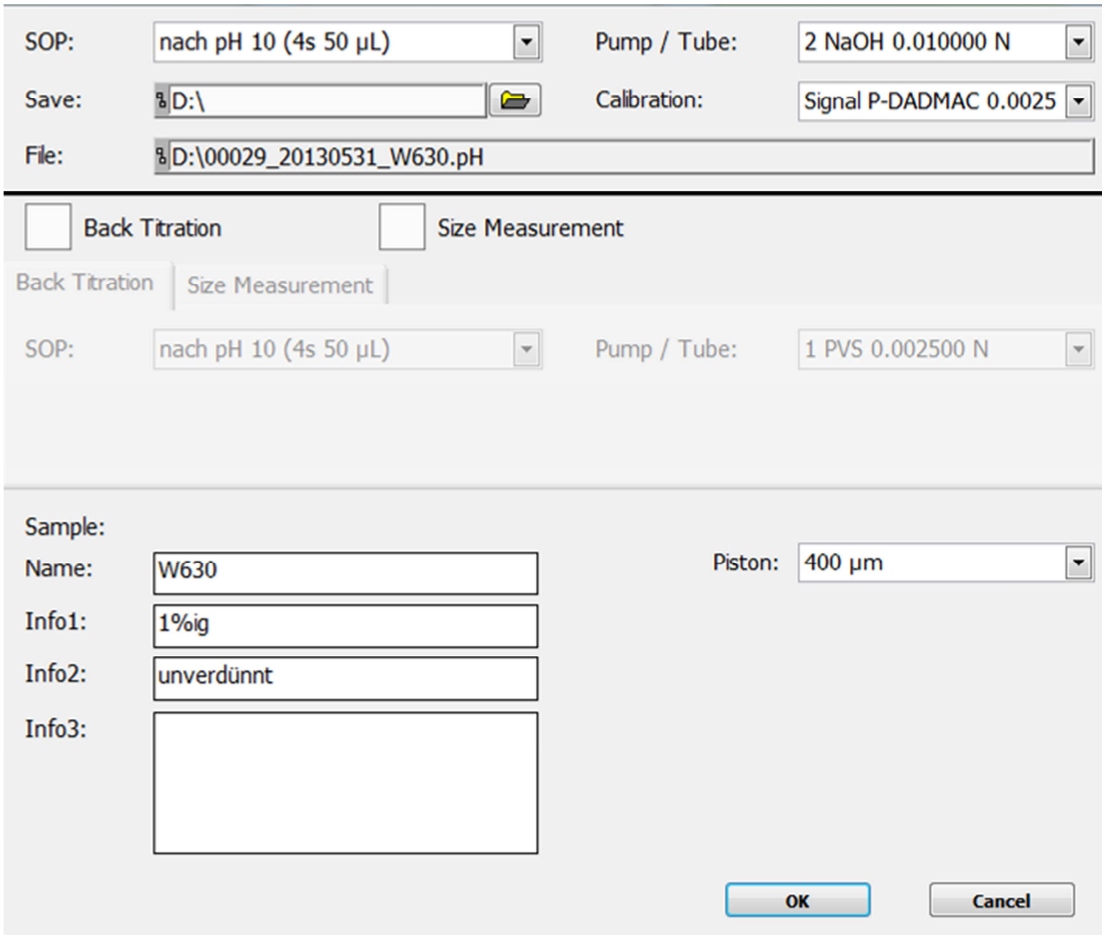
Please enter the sample information.

First of all, the sample name should be entered. The sample name forms an important part of the file name.



Attention!
Without an entered sample name the titration cannot be started!
The file name is synthesized in the following way: A run number and the date is set in front of the sample name.

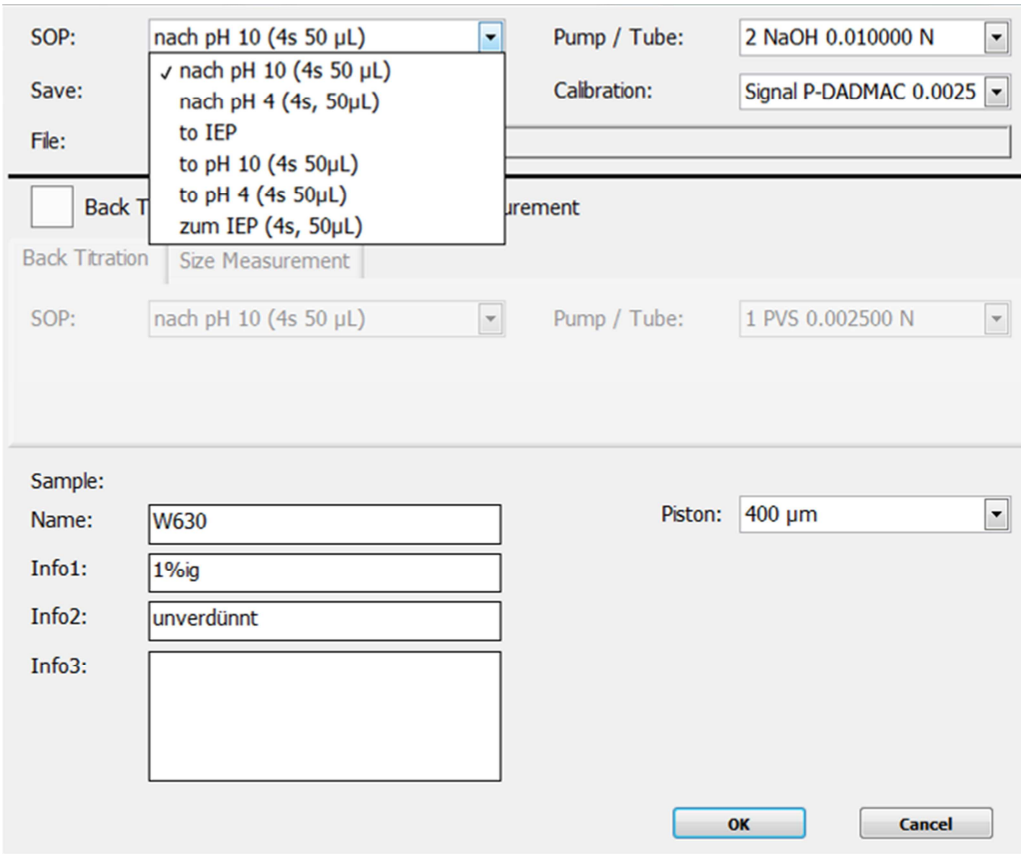
Further sample information like start pH-value, concentration, dilution or other comments may be entered into the fields "Info1" to "Info3".



In chapter 4.5.1 the extended measurement modes are explained:
Back Titration, Size Measurement and Calculations.

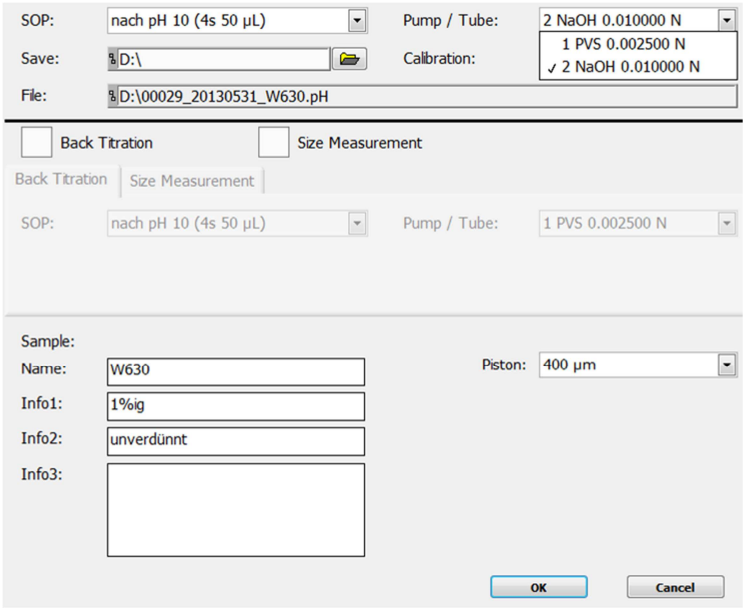
After the file name is assigned, the desired SOP may be selected. Here, a titration to pH 10 is selected.

Notice
In the field „SOP" nothing but methods are displayed, which are already programmed and importantly, which are associated to the selected titration type.



In the next step the titration solution (Name of titrant/Pump #) is selected.

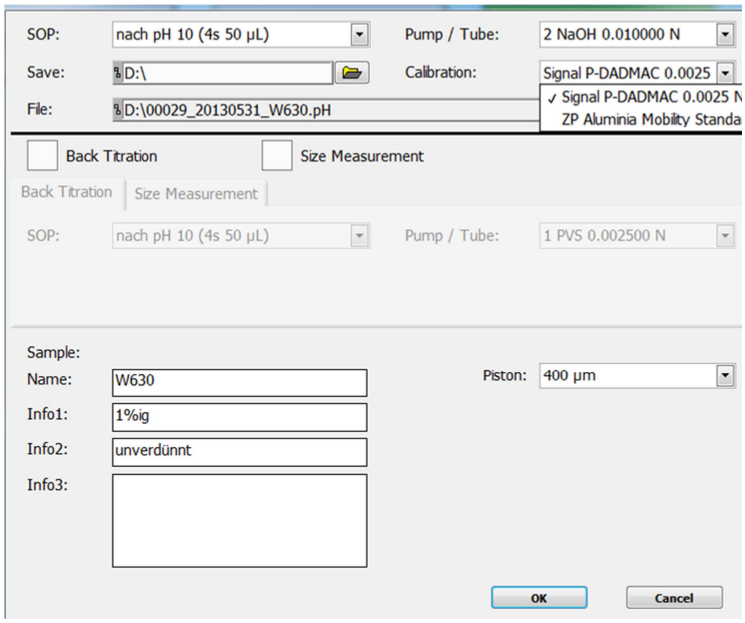
Notice
Only if titration experiments are intended, it is required to enter the titration solutions. Pure potential experiments (without titration) do not need an entry. No matter what is written into the box, it will not be documented having chosen one of the potential measurement types.



After selection of the titration solution the calibration type has to be chosen. Several options are open:

Signal: Calibration to +1200 mV with 0,001 N P-DADMAC cationic polymer solution (analogue to the StabiSizer® streaming potential)

ZP (zeta potential): Calibration a) to +50 mV with the Microtrac mobility standard suspension or b) to any other commercially available zeta potential standard particle dispersion or c) to +73 mV with WAKO 0.1N P-DADMAC solution.

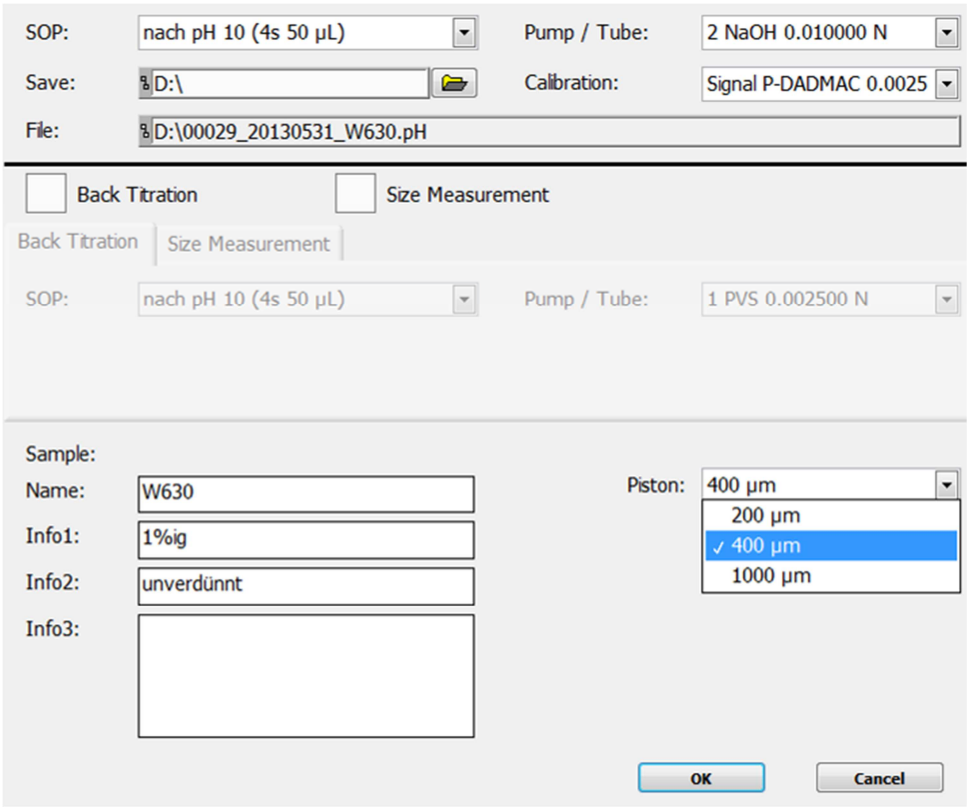


For polymer samples it is preferable to calibrate with P-DADMAC, a cationic polyelectrolyte. For particle dispersions a calibration with a particle standard suspension is recommended.
More information on calibration procedures can be found in the chapter 7. "Calibration of the Stabino®".

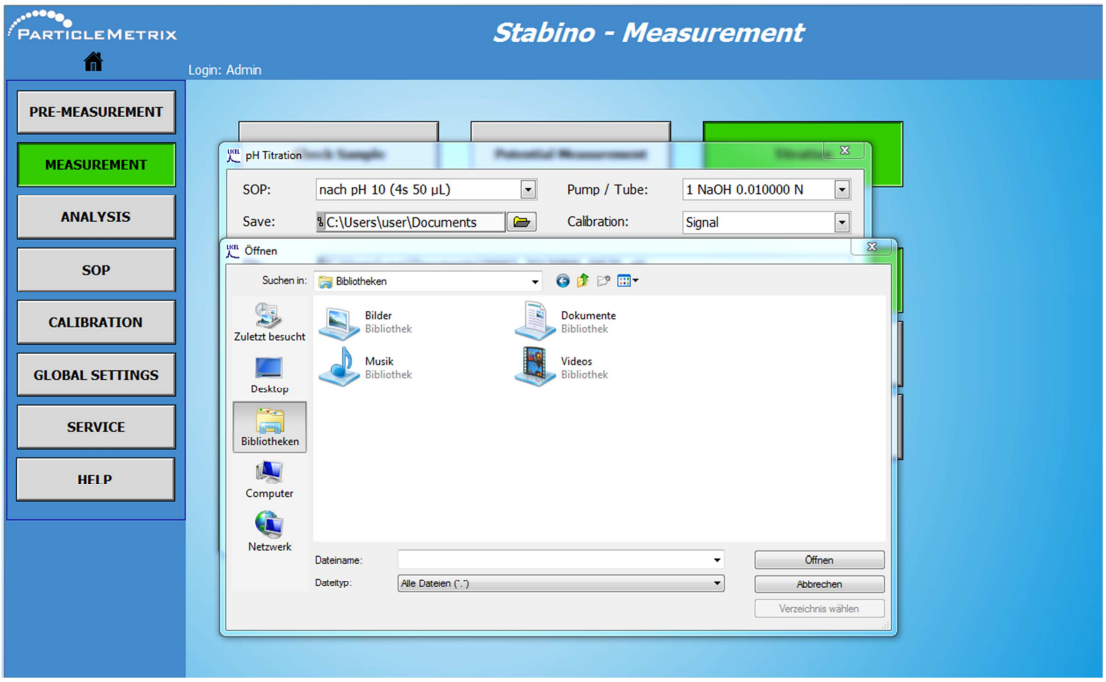
The last step of the settings requires the entry of the piston – "Piston".

Attention!
It is important to insert the correct piston. The height of the measured potential depends on the gap between piston and cylinder.

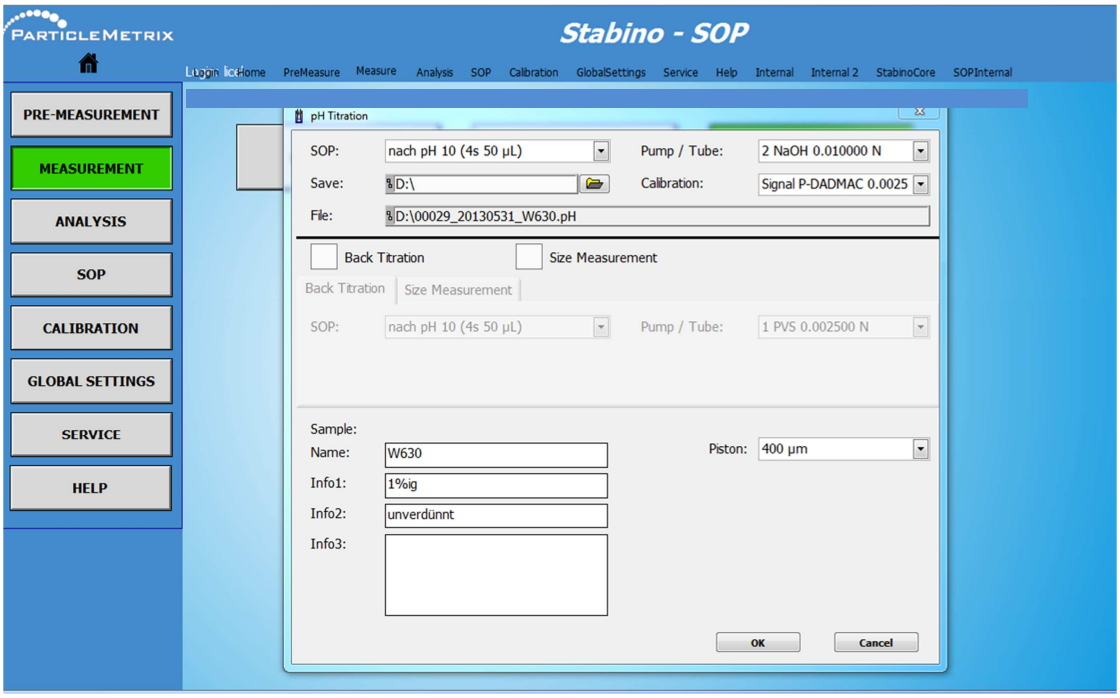
Note
A small gap causes a big potential. Rule of thumb: Half the gap doubles the measured signal.



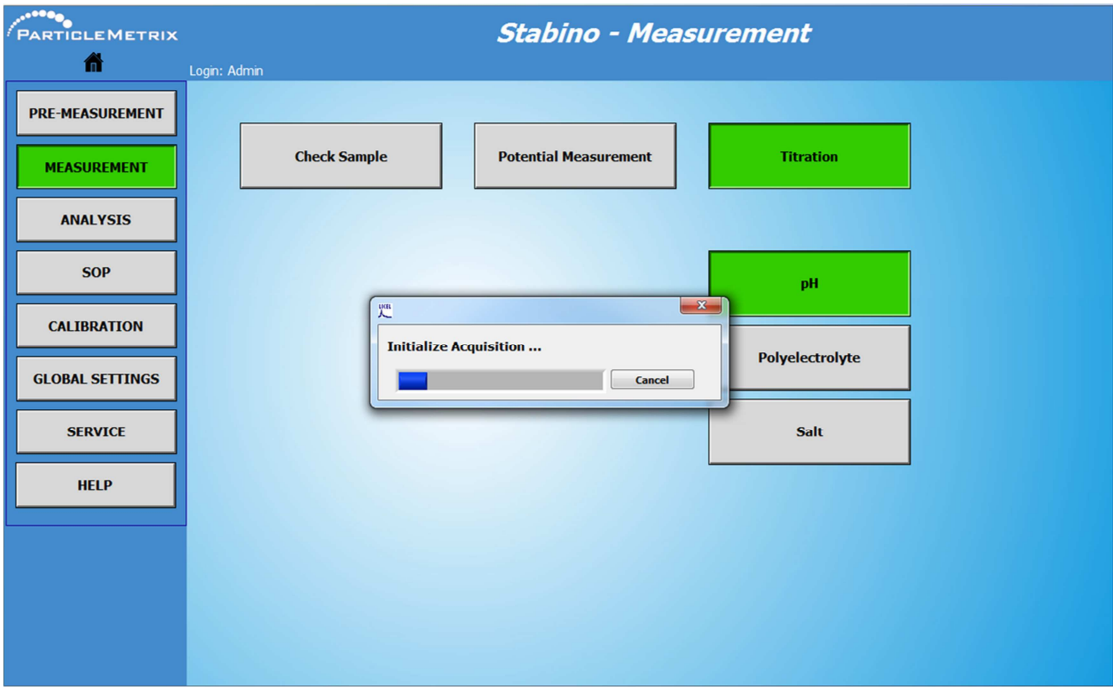
The path and memory allocation can be set by "Save".



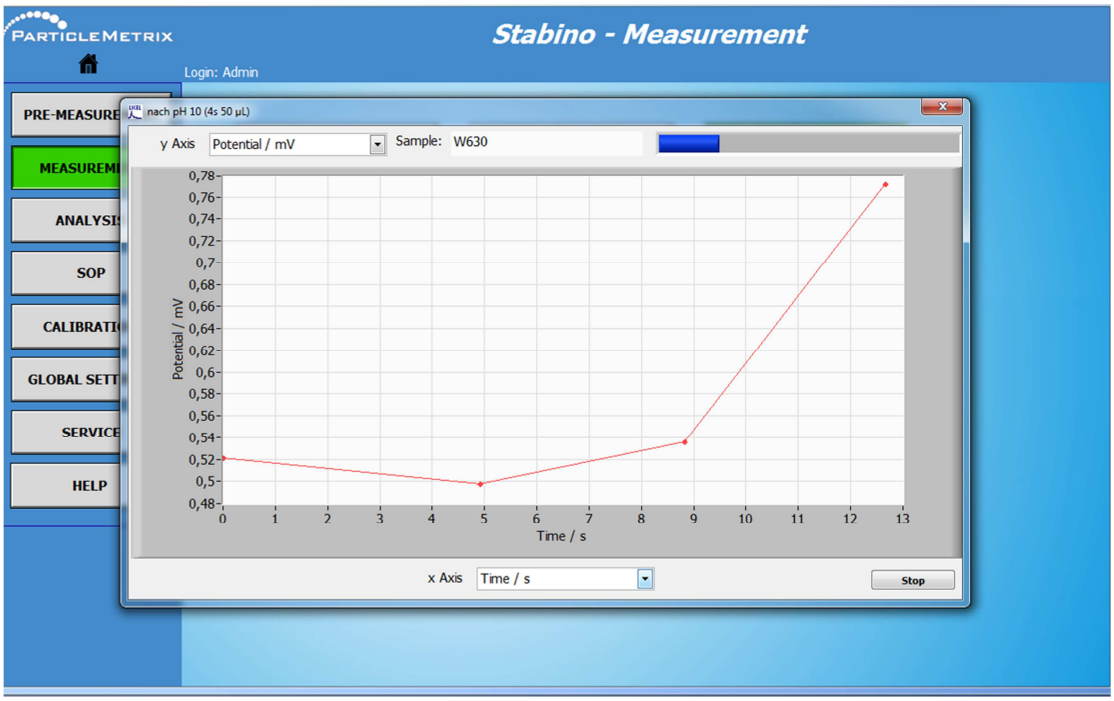
After having carried out all settings the measurement can be started with “OK”.



Before the actual measurement starts, the sample is mixed for 30 sec by movement of the piston.



After 30 seconds of mixing action, the measurement window opens, where the signal can be followed „online" during titration.



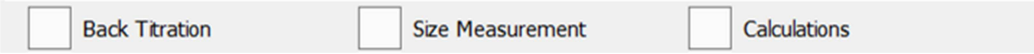
On top of the y-axis and below the x-axis (see red arrows below) you find pull-down buttons for the selection of the data presentation: potential versus pH, volume, time etc.

As soon as the measurement is finished, the software jumps to the analysis menu. Please read chapter 5 for more information on analysis possibilities.

If you start a further measurement of the same type (example pH titration), the new curve is overlaid to the old ones. If a measurement of a different type (than displayed before) follows, only the last measurement will be presented.

4.5.1 Extended measurement settings

After having selected the measurement method, another dialogue "sample input" allows an extended choice with "Back Titration", "Size Measurement" und "Calculations".

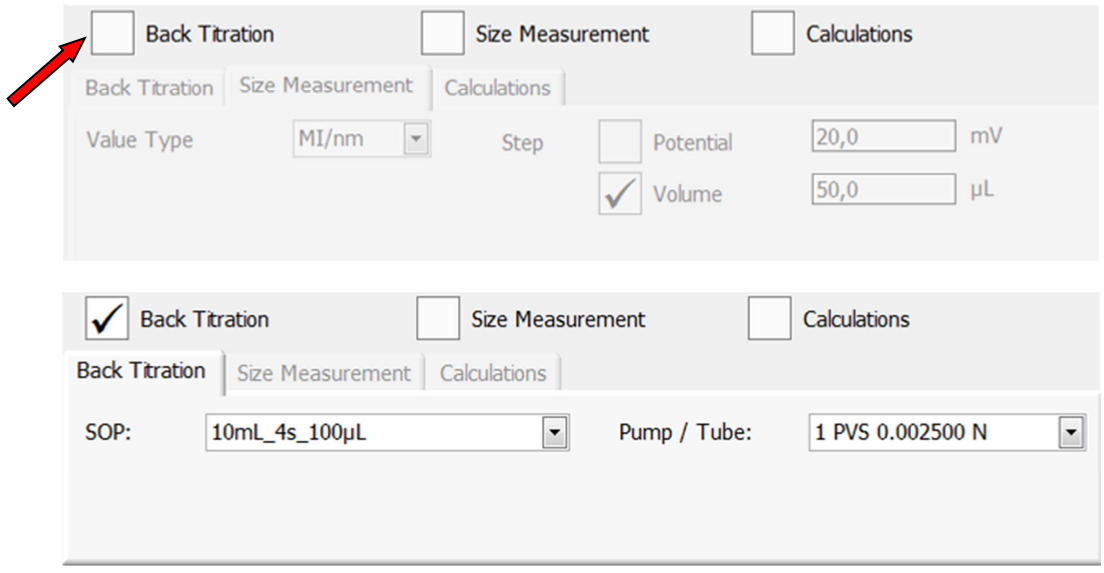


Back Titration	With "Back Titration", a second titration is programmed to follow. Reasons to program a "back titration" could be to titrate back into the reverse direction to study hysteresis effects.
Size Measurement	180° DLS Size measurement via the Microtrac Flex Software. For the size measurement, the movement of the piston is stopped at defined stop points. The size data are then entered manually into the Stabino Control software.
Calculations	Calculation of the ionic activity of polymers or of the specific charge density of particles

The following section describes which extended measurement methods are available for what kind of measurement method.

Measurement method:	Possible further measurement settings:
Current Status	Size Measurement
Kinetics	Size Measurement
pH	Size Measurement, Back Titration
Polyelectrolyte	Size Measurement, Back Titration, Calculations
Salt	Size Measurement

Select the method with a click (red arrow example back titration). The corresponding menu is opens to enter relevant parameters.



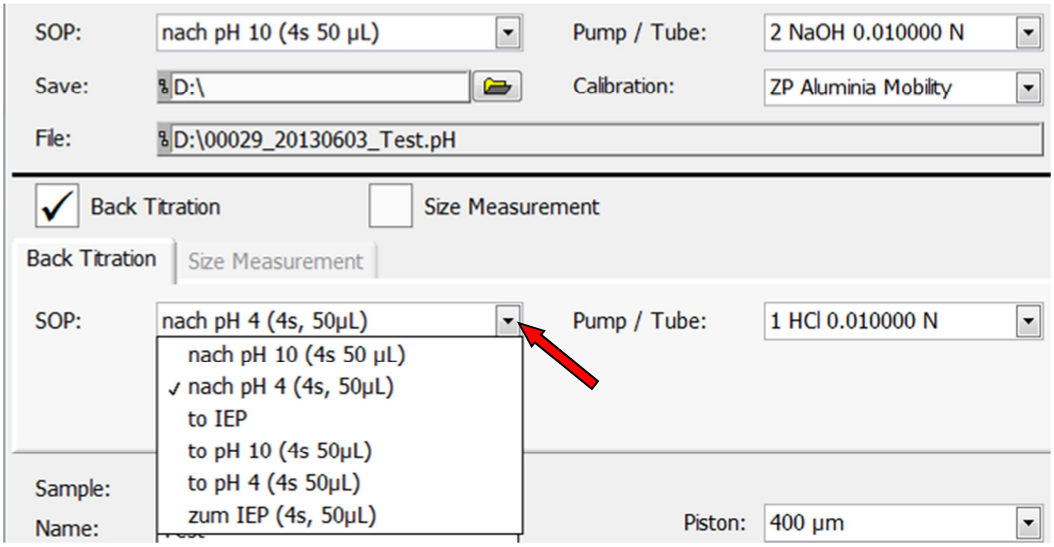
The programming and performance of these menus is described in detail below. One or all three extended programs can be selected.

Back Titration:

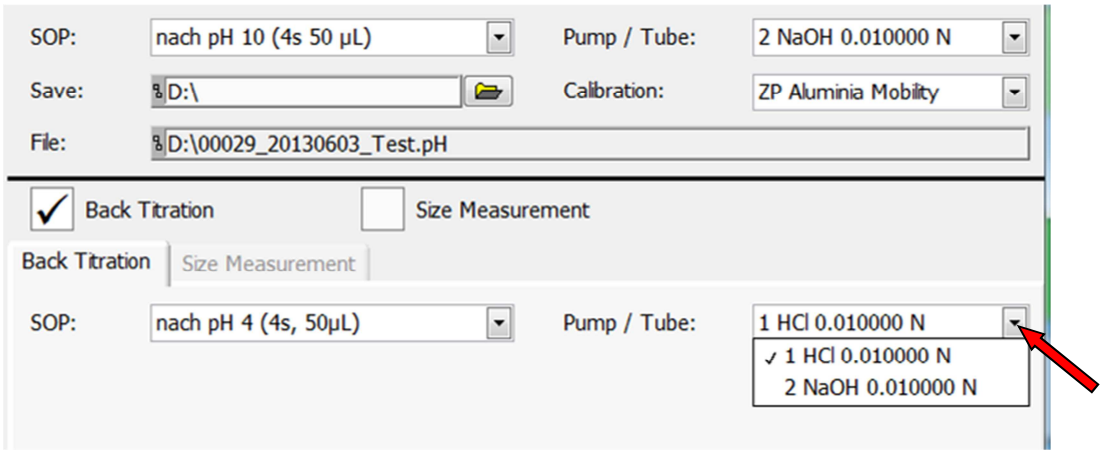
Under a "back titration" a second titration is started atomatically after the first one. In this example the first titration runs from pH = 4 to pH = 10, the second from pH = 10 to pH = 4.

Please note:
The measurement data of the 2 titrations are stored as 2 titration files. In the analysis mode, the files can be opened individulaly.

Open the drop down menu (red arrow) to select the SOP for the "back titration". The routine is the same as with one single titration.



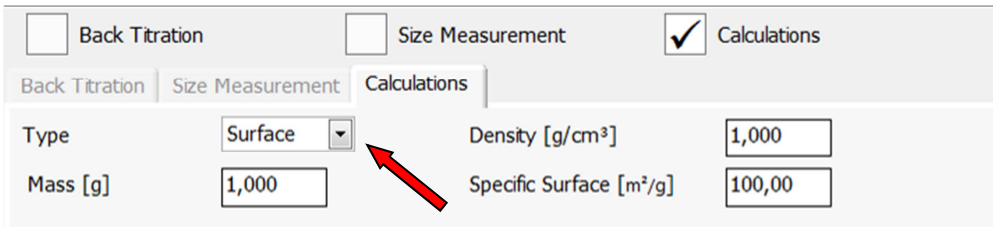
As with a single titration, the titrand solution and the corresponding pump (red arrows) have to be selected.



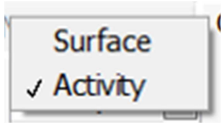
Calculations

Two drop down menus are available under "Calculations" (red arrow).

Please note:
The concentrations of the titrand solutions, which are needed for the calculation, are already given in the "PRE-MEASUREMENT".



Choice between "Surface" and "Activity":



With "Surface", following calculation is offered:

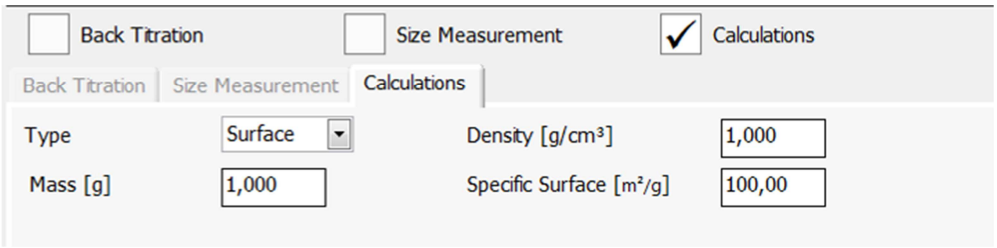
- Charge consumption to reach the zero point of charge
- Specific charge density
- Weight specific charge

With "Activity", following calculation is offered:

- Charge consumption to reach the zero point of charge
- Charge consumption per liter sample
- Weight specific charge
- Specific charge per mole sample

To calculate surface charge (Type: "Surface") following data are needed:

1. Sample weight (Mass)
2. Density of the sample
3. Specific surface of the sample

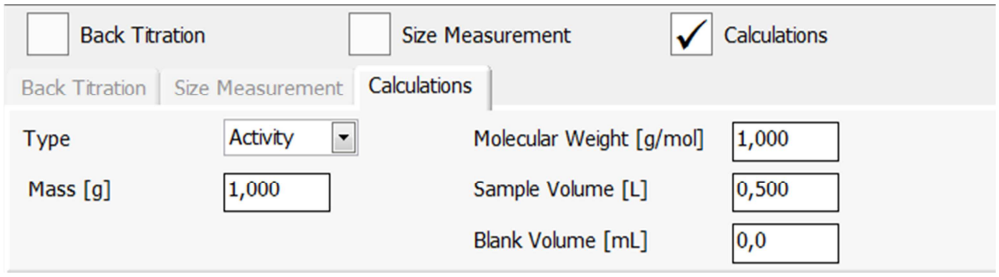


When started, the experiment runs like a standard charge titration.

For the calculation of the activity of polymers following data are needed:

1. Weight of the sample (mass)
2. Molecular weight of the sample
3. Sample volume
4. Consumed volume of titrant solution for the solvent (Blank Volume)

Please note:
The consumed volume of titrant solution for the solvent is deduced from the consumption of charge by the sample. The so calculated activity is more accurate. If the blank volume is not known or if it was not measured, the "0,0" can be left in the value field.



After entering the necessary parameters, the measurement is started. It runs like a standard charge titration.

Attention
It is not possible to do a recalculation with paramters entered after the measurement. Parameters only are accepted when entering before measurement.

Size Measurement

Under this menu size can be measured with the dip-in sensor of the optional 180° DLS NANO-flex instrument and manually entered into the potential measurement / titration.

Please note:
For the size measurement in the Stabino sample cell you require a NANO-flex instrument which is based on the 180° heterodyne DLS dynamic light scattering method. The results are typed into the Stabino control software manually.

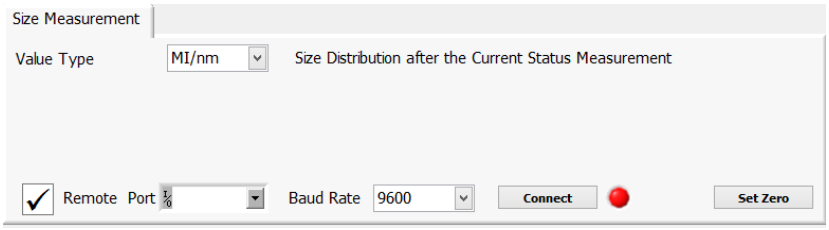
Following size quantities can be selected from the size distribution measurement, whereby MI, MV, MN und MA can be seen as mean values (see separate manual of the NANO-flex):

MI: Intensity weighted size
MV: Volume weighted size
MN: Number weighted size
MA: Area weighted size
D50: D50-value of the size distribution

To each measurement method specific stop points are attributed.

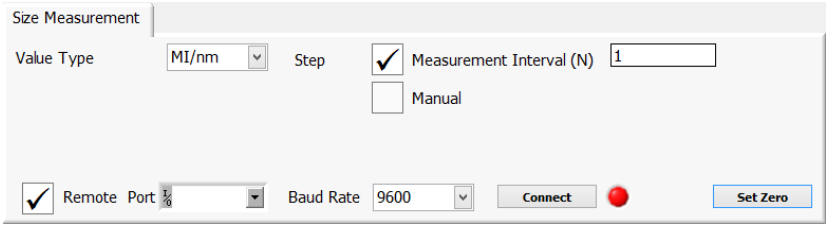
Current Status

The size measurement is performed at the end of the potential measurement.



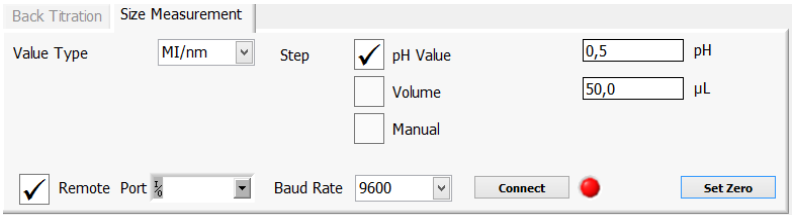
Kinetics

1. The size measurement is performed after each N th measurement interval. Example: 1= after each interval. 2= after each second interval.
2. Manually entered measurement interval: The user defines when the size measurement should be performed.



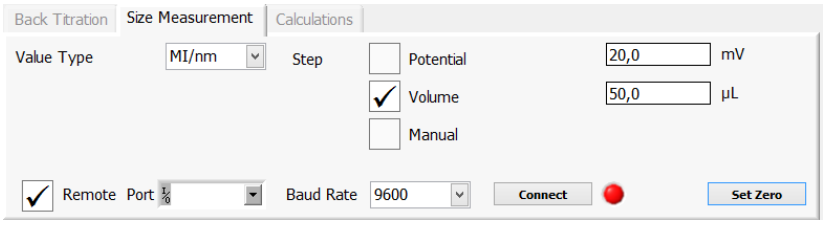
pH

1. The size measurement is started after each defined change of the pH value.
2. The size measurement is started after each defined added volume interval.
3. Manually entered measurement interval: The user defines when the size measurement should be performed.



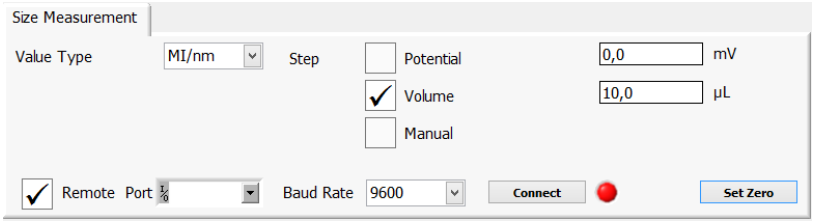
Polyelectrolyte

1. The size measurement is started after a defined change of the potential.
2. The size measurement is started after each defined added volume interval.
3. Manually entered measurement interval:



The user defines when the size measurement should be performed.

Salt



1. The size measurement is started after a defined change of the potential.

2. The size measurement is started after each defined added volume interval.

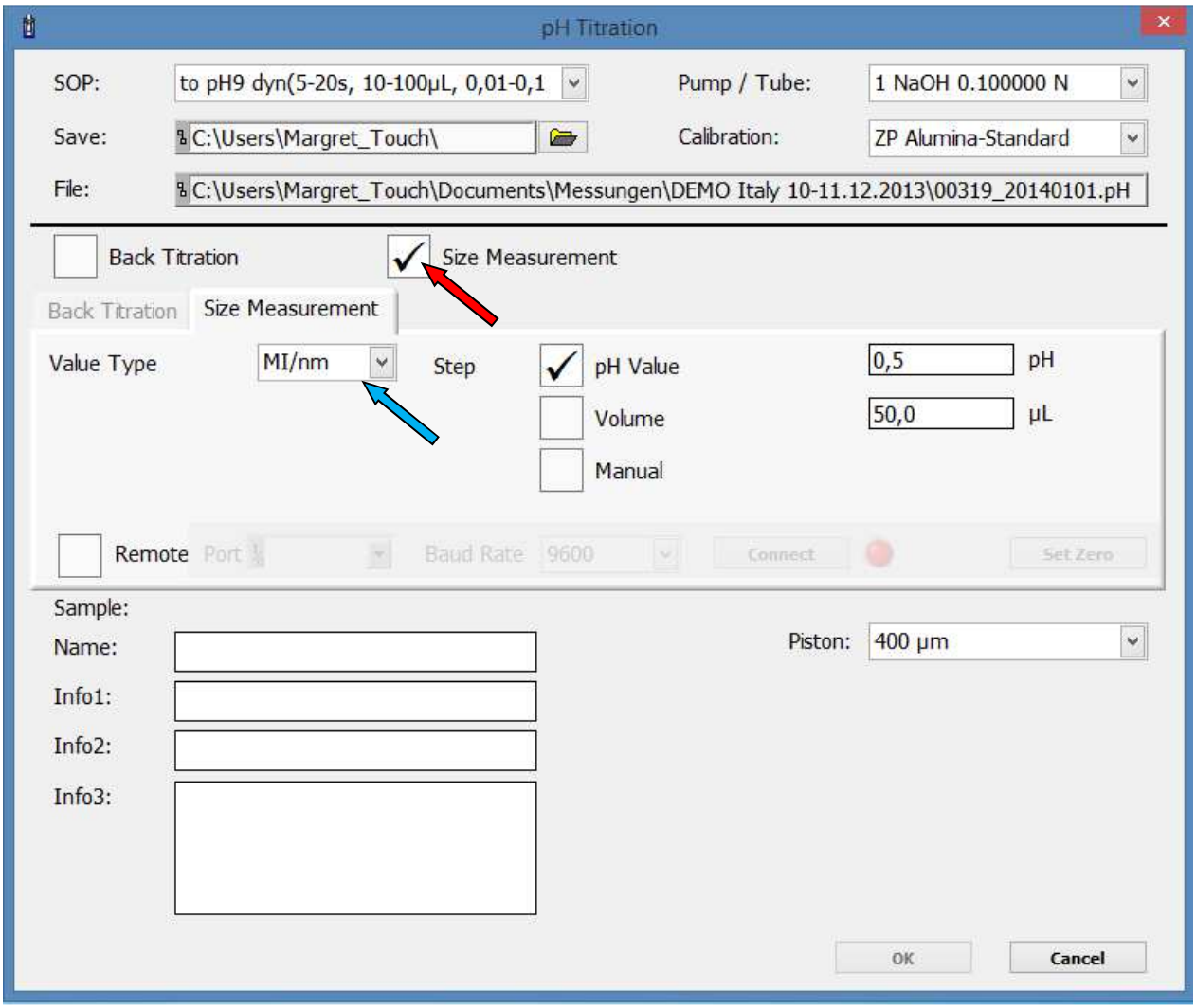
3. Manually entered measurement interval: The user defines when the size measurement should be performed.

A combined particle charge and size measurement **without Remote Control** is performed as follows:

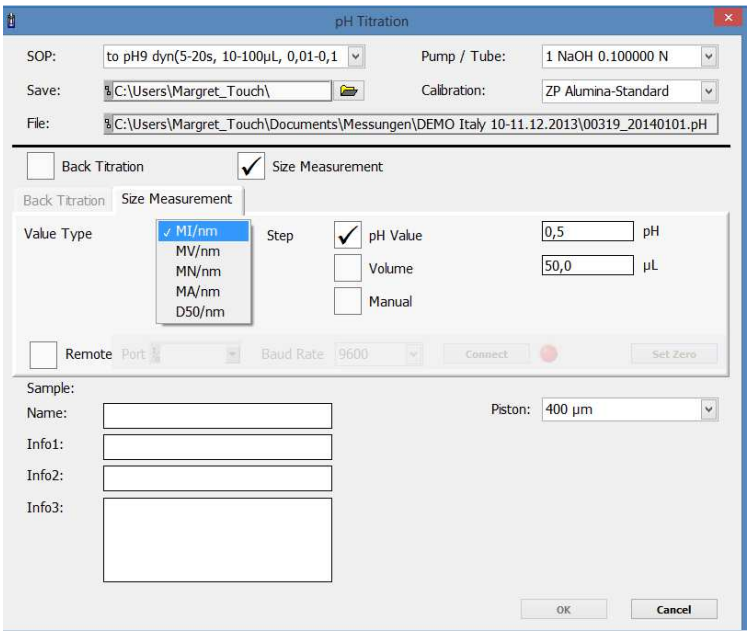
Please note

Before starting a combined charge & size experiment a setzero / blank measurement with NANO-flex on the dispersion medium is required.

At first, activate the size measurement (red arrow).



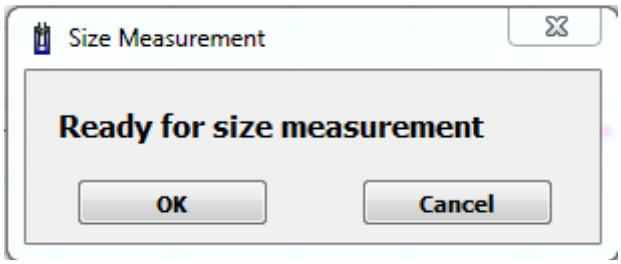
Second, specify the kind of size presentation in the data (blue arrow). In the opened drop down menu all choices are listed.



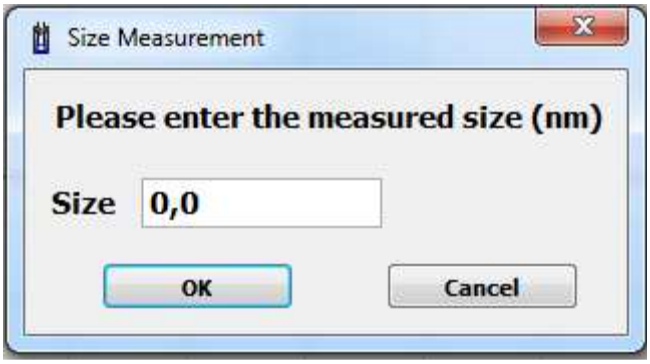
Third, chose the interval of alteration, after which a size measurement is required.

Now start the measurement.

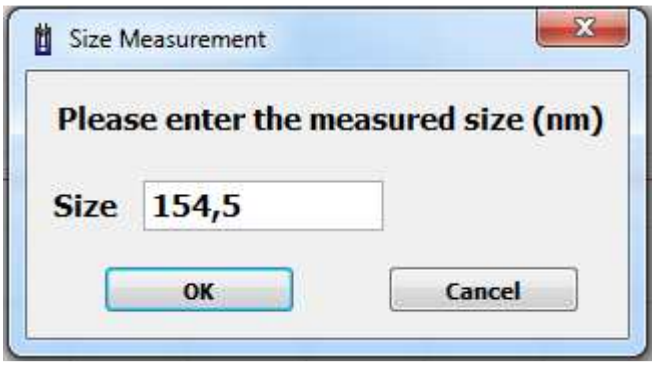
After reaching the conditions for the first measurement stop for the size determination, following windows opens.



Klick „OK“. Perform a size measurement with the MICROTRAC FLEX software (for more details see Operation Manual of FLEX).
After the size measurement is finished, window opens to enter the size opens.



Please enter the size, which was measured with the NANO-flex and confirm with “OK”.

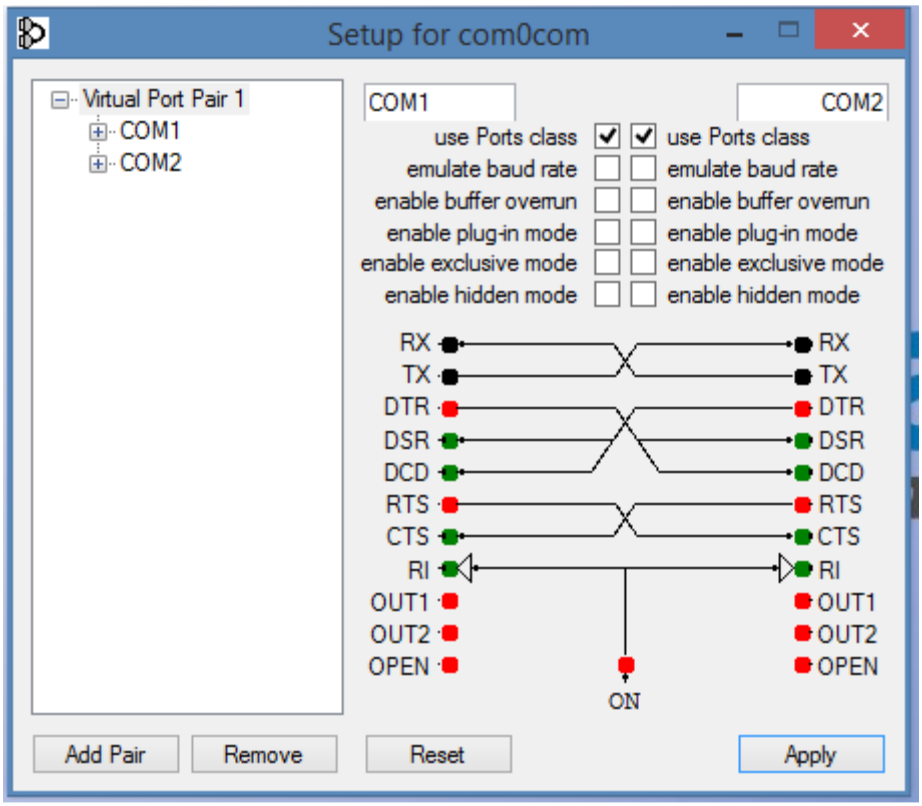


The titration is now continued until the next stop interval is reached. A new size measurement is required. This procedure is repeated until the titration end point is reached.

A combined charge and size measurement under **Remote Control** is performed as follows:

Note
Remote Control is only applicable when the instrument NANO-flex is connected and the Microtrac FLEX software activated.

Before the measurement can be started, a COM – COM emulation has to be done. In addition, two COM ports have to be established. As an example: ***COM1*** and ***COM2***.

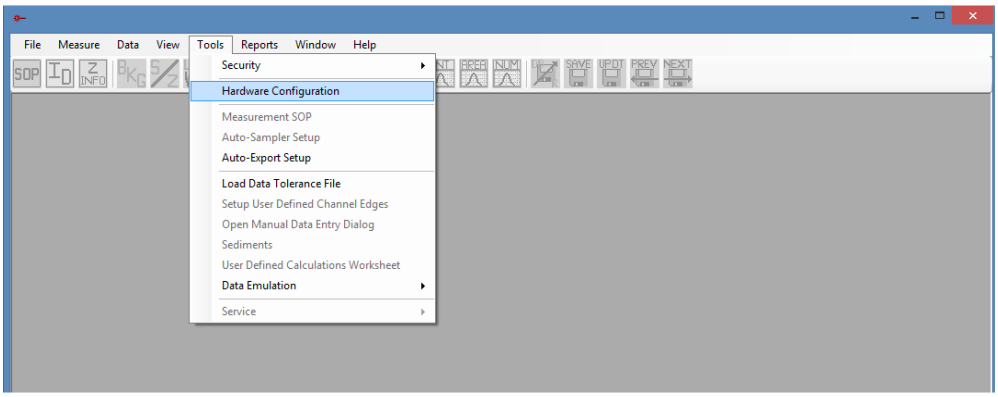


Note
COM1 is the COM-port of the NANO-flex and ***COM2*** is the port of the remote control.

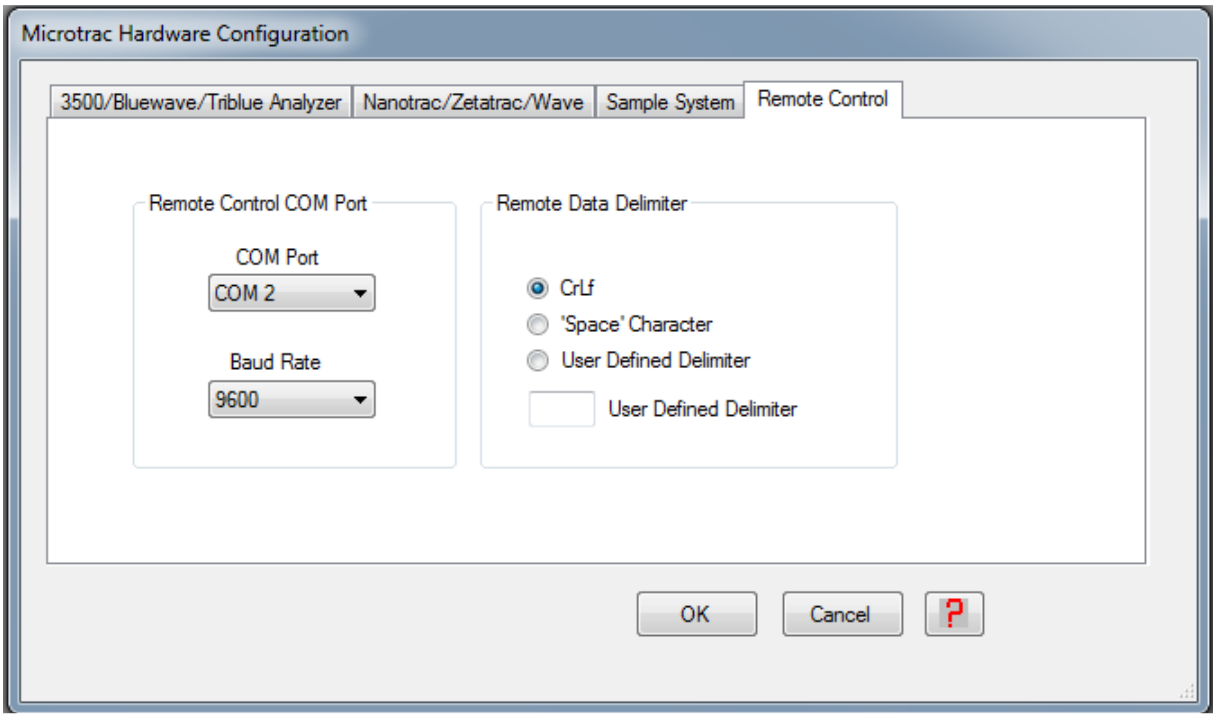
Note
For the installation of the COM-COM Emulation please read the instructions about the emulation software.

In the following you start the Microtrac Flex Software.

Please go to Tools → Hardware Configuration:



Following window opens. Please select REMOTE CONTROL and choose in ***COM2*** and 9600 Baud Rate, as an example.



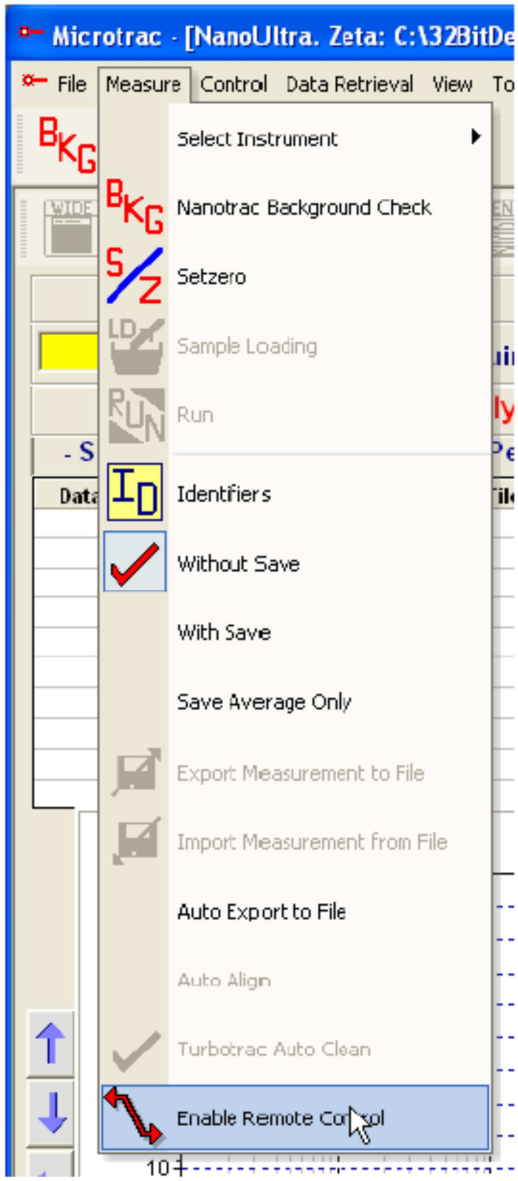
Select ***COM2*** and Baud Rate 9600, select a data base and activate REMOTE.

Note
The setting of the COM-port in the Microtrac Flex Software has to be done only once. These data are stored and have to be changed only in case when a new COM port has been selected.

In the following open a data base and select the appropriate SOP for the size measurement.

Note
For the correct operation of the Flex Software please read the manual of the Flex Software and the short instruction for the NANO-flex.

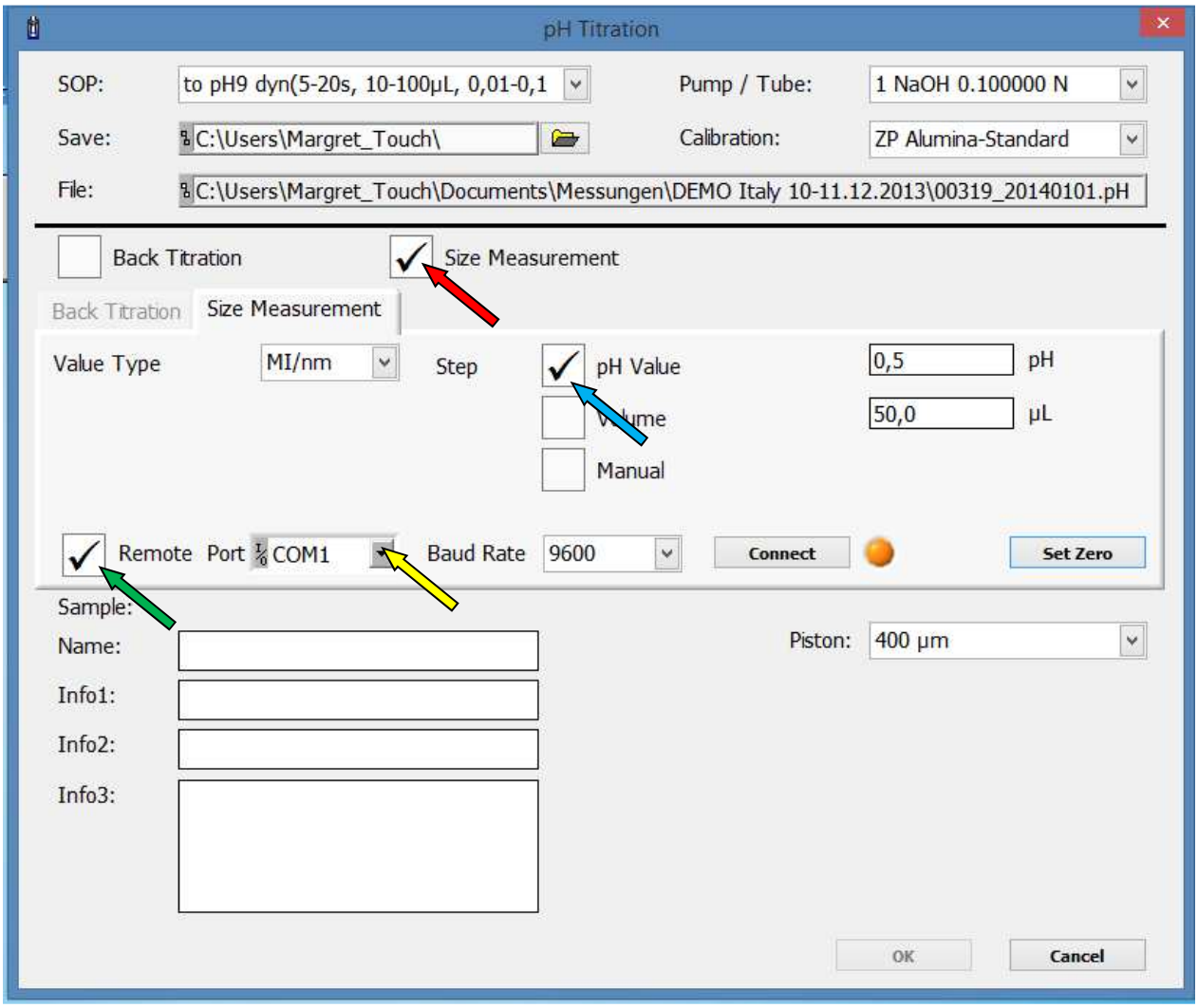
After opening the data base and selection of the SOP please click on MEASURE and select ENABLE REMOTE CONTROL.



Now all settings in the Flex software are completed. Now you can continue using the Stabino Control Software.

Note
The SOP timing has to be set to the following parameters: Setzero Time = 30 sec Meaurment Time = 30 sec, Number of Runs =1

Now activate the size measurement in the Stabino software (red arrow).



Select the VALUE TYPE of the size in the drop down menu (MI or MV or MN).

Select the STEP INTERVAL for the size measurement.

Activate REMOTE MODUS (green arrow) and select the COM port (yellow arrow) (here ***COM1***). The Baud Rate has to have the same value as in the Flex software.

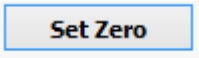
Note
Select the COM port which controls the NANO-flex.

The control light next to the button "Connect"- still shining in red – starts blinking in yellow-green.

Following colors are available:

Bright green	No error, connection successful
Orange	Error message by the Microtrac Flex Software. Please check the error message in the Flex software.

Red	No communication with the Flex Software or communication error.
Dark green	Unknown error.

Please perform SET ZERO by clicking the button 

and follow the displayed instructions.

Note
Before the combined charge / size measurement is activated the SETZERO/BLANK measurement with the NANO-flex has to be performed on the dispersion medium.

Start the measurement as usual.

The charge titration is started and automatically stopped at the first pre-programmed size measurement point. Every time, when the pre-programmed points are reached, the piston stops and the size measurement is performed. After finishing the size measurement, the charge titration starts again. This procedure is repeated until the end point of the titration is reached.

Attention
The size measurement delays the duration of the titration.

Attention
If an error occurs during the size measurement the titration is stopped.

5. Analysis mode

5.1 Installation of the software on other working stations

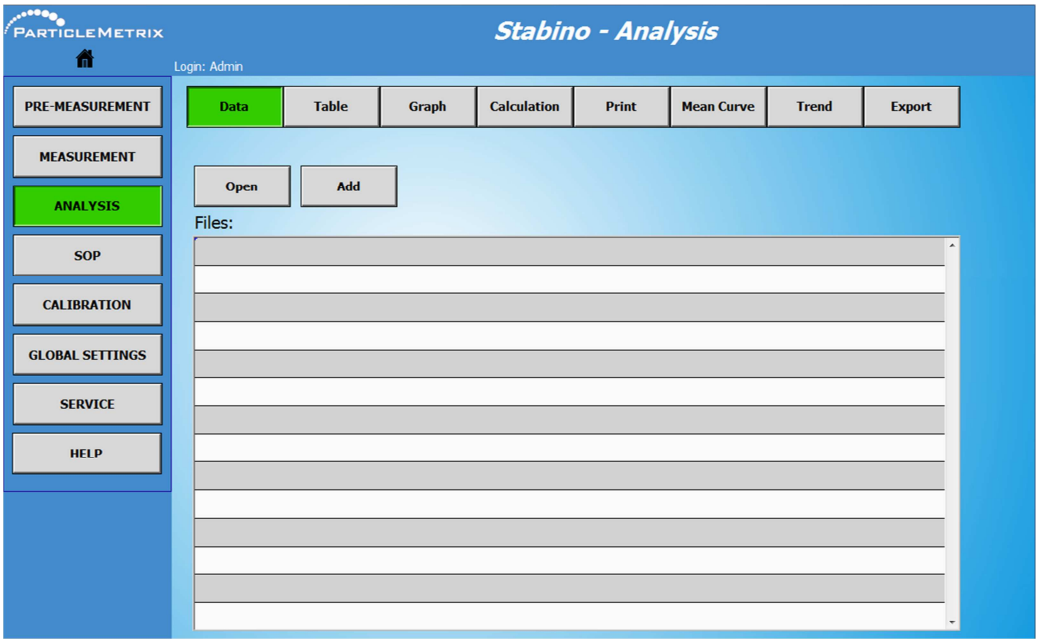
The software is not licensed and can therefore be installed on other PCs. Follow the description in chapter 3.2.3. If the software is opened without TCP/IP-connection to the Stabino®, it starts with the "Analysis" mode.

Without connection to the Stabino® the software can only be used in the "Analysis" mode. Only the functions "Analysis", "SOP", "Global Settings", "Service" and "Help" are responsive.

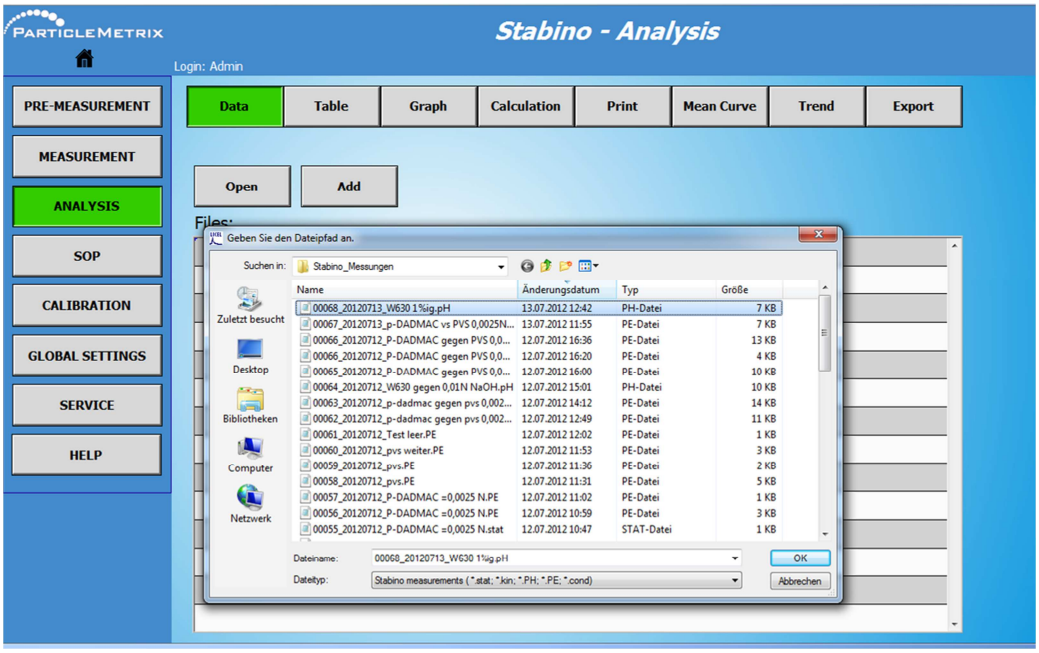
Note
For the installation of the software on your PC you need administration rights.

5.2. Data

Measurement data can be opened in the window "Data". Click the button "Open".

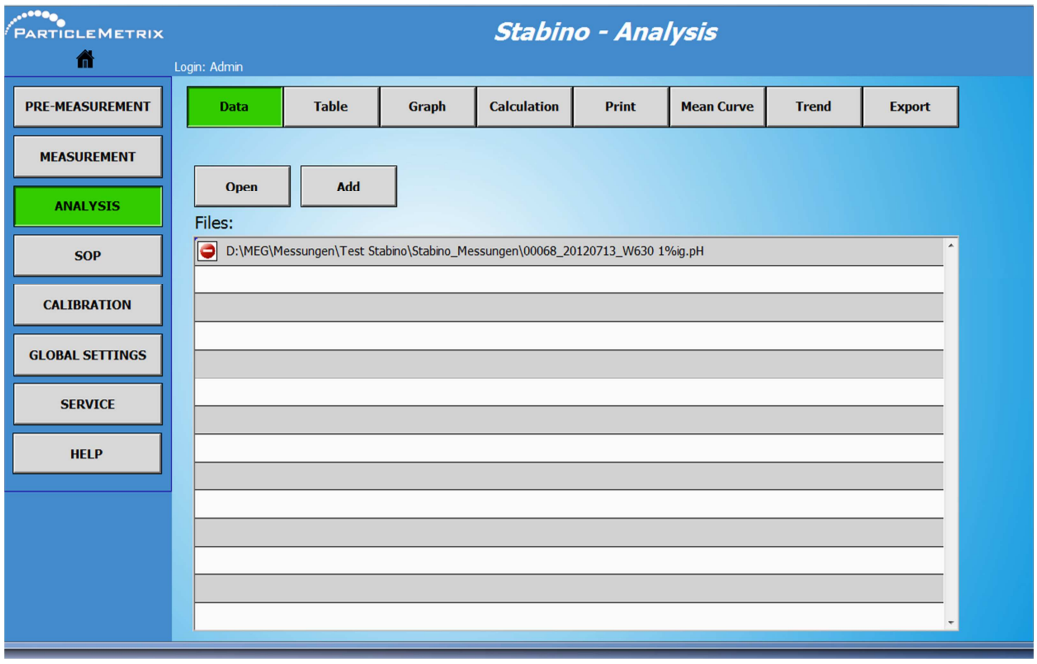


A dialogue window opens to select the folder with the data you want to display.



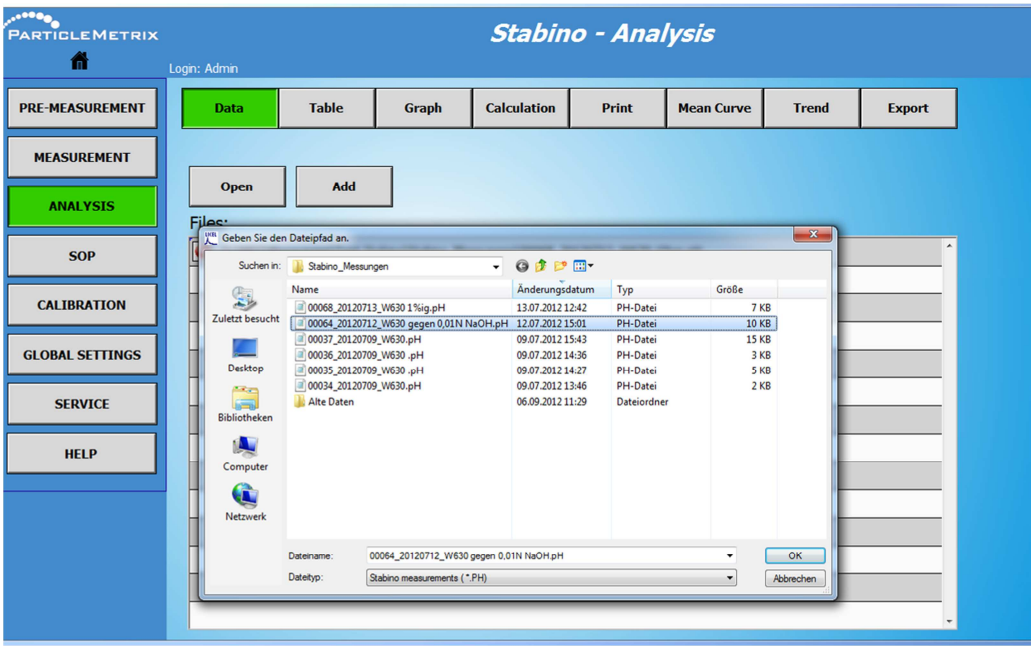
Please select the desired file and click “OK”.

The selected file will be indicated in the window “Files”.



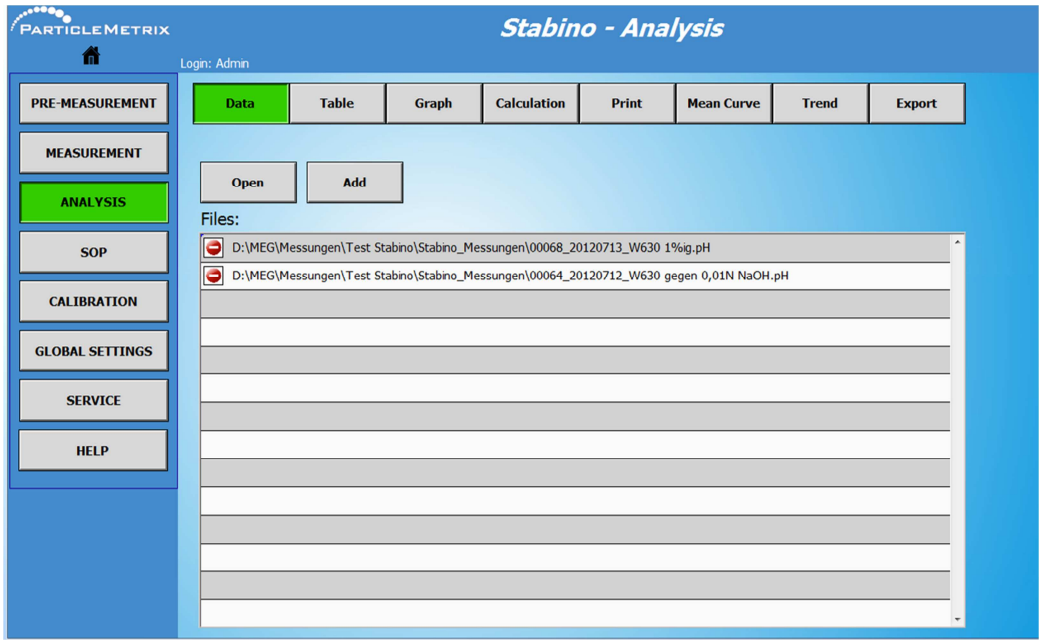
In order to open further data for comparison or for overlay purposes, click “Add”. Again, the window opens where you can select further data.

Note
With the function „Add“, only measurement data of the same type can be opened. In the repertory window only data of the same type are indicated.



Select your data and click “OK”.

The selected file is now indicated in the window “Files”.



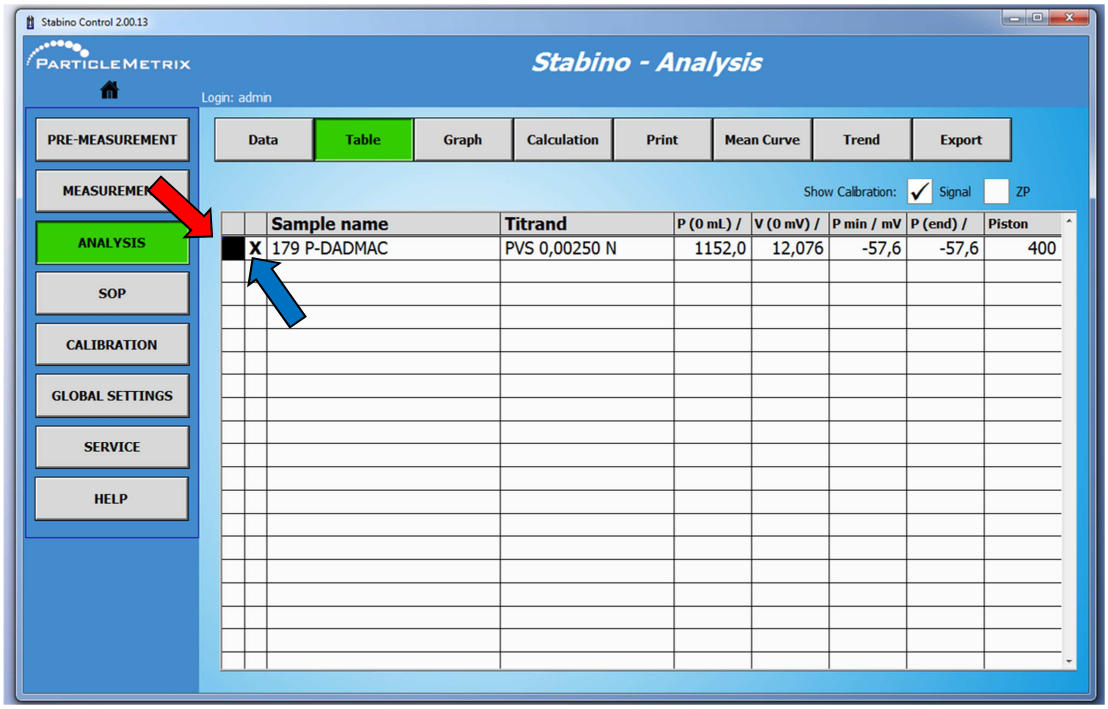
To close an open file press on the  symbol.

Note

If you want to open a file of another measurement type, click “Open” and select the corresponding file. All other data shall be closed automatically.

5.3. Tabular presentation of results

In the menu "Table", all important measurement data and parameters are presented. The first 2 columns are assigned to the names of the sample and the titrant. By checking the boxes for "Signal" or "ZP" beside the "Show Calibration" field, the operator can decide, if the result for the potential will be presented as streaming potential (Signal) or as zeta potential (ZP).



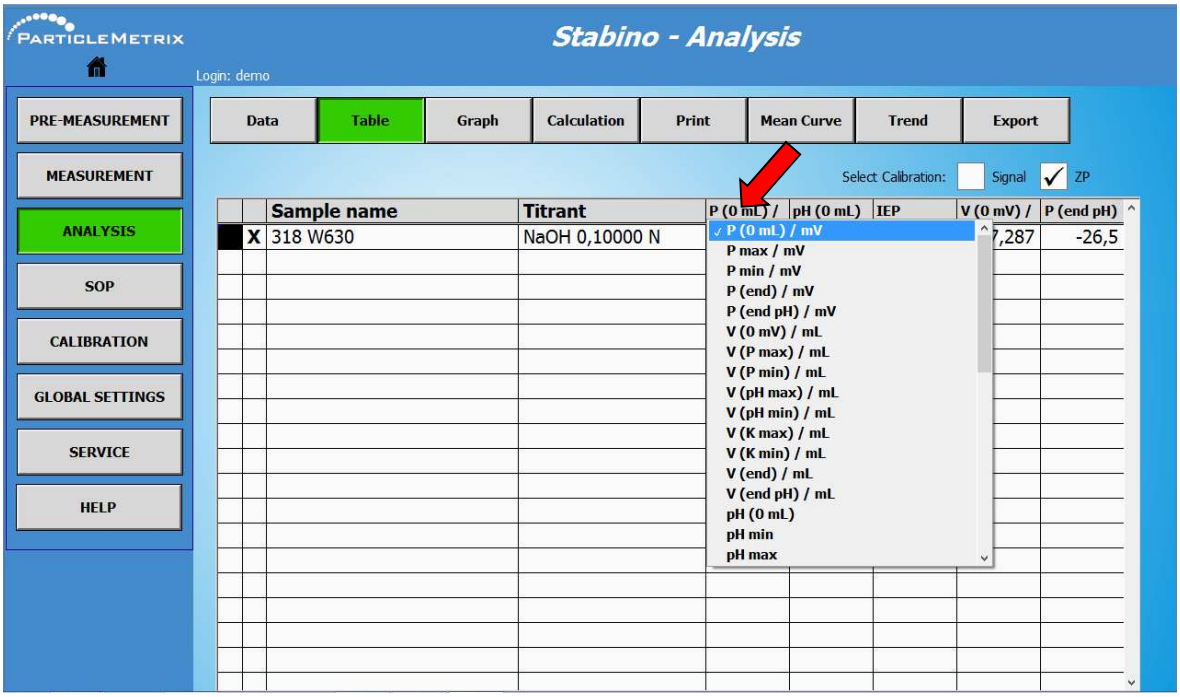
By clicking the "black" field (red arrow), the color of the graph belonging to the sample name can be selected.

Following dialogue window opens:

Here you may select the desired color. The order of the colors can be preselected under the menu „GLOBAL SETTINGS" → Colors.

Should the selected sample be displayed as a graph or not, klick „X" (blue arrow).

In each of the next 5 coloumns one of the listed measurement parameters or result data can be chosen for being displayed in the record. To do this, click on top of the column (red arrow).



The assortment menu opens with following selection possibilities:

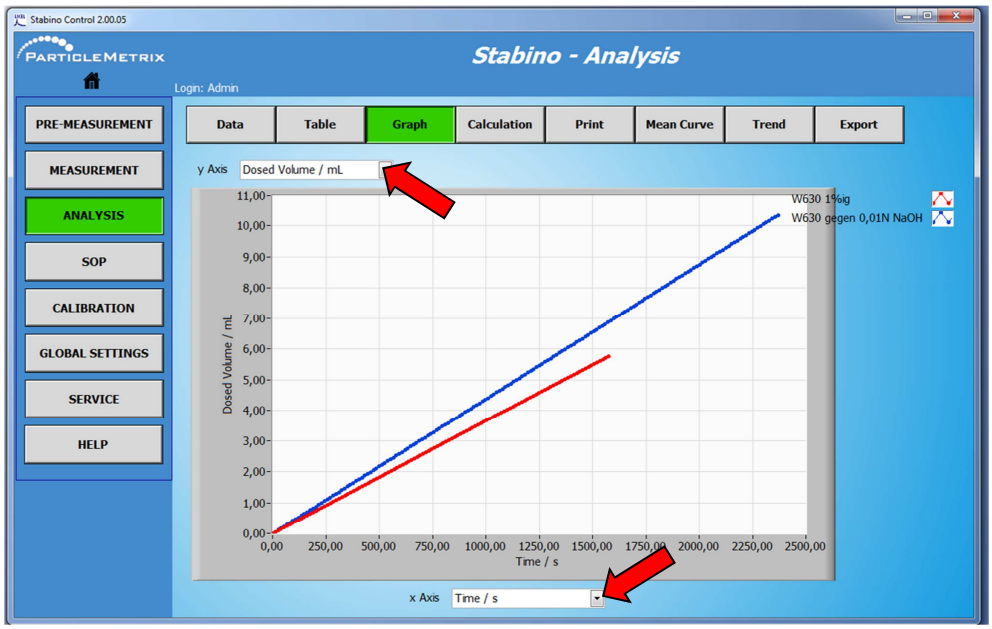
P (0 mL) / mV	Value of the starting potential
P max / mV	maximum potential
P min / mV	Minimum potential
P (end) / mV	Last measured potential
P (end pH) / mV	Measured potential at the end point pH
V (0 mV) /mL	Consumption of titrant solution to the zero point of charge
V (P max) / mL	Consumption of titrant solution to the maximum potential
V (P min) / mL	Consumption of titrant solution to the minimum potential
V (pH max) / mL	Consumption of titrant solution to the maximum pH value
V (pH min) / mL	Consumption of titrant solution to the minimum pH value
V (K max) / mL	Consumption of titrant solution to the maximum conductivity
V (K min) / mL	Consumption of titrant solution to the minimum conductivity
V (end) / mL	Total consumption of titrant solution
V (end pH) / mL	Consumption of titrant solution to the end-pH-value
pH (0 mL)	Value of the Start-pH
pH max	Maximum pH-value
pH min	Minimum pH-value
pH (P max)	pH-value at the maximum potential
pH (P min)	pH-value at the minimum potential
IEP	Isoelectric point
K (0 mL) / S cm^{-1}	Value of the conductivity at start
K (0 mV) / S cm^{-1}	Conductivity at the point of zero charge

K max / S cm^{-1}	Maximum conductivity
K min / S cm^{-1}	Minimum conductivity
K end / S cm^{-1}	Last measured conductivity
Size Type	Indicates the type of the displayed size value
Mx max / nm	Maximum measured size
Mx min / nm	Minimum measured size
Mx (start) / nm	Size at starting point
Mx (end) / nm	Size at the end of the measurement
ABS (V (P min) – V (0 mV)) / mL	Consumption of titrant from the minimum to the zero point of charge
ABS (V (P max) – V (0 mV)) / mL	Consumption of titrant from the maximum to the zero point of charge
Date & Time	Date and time of measurement
Piston	Indicates which piston is used
Info 1	Indicates the text written under Info 1
Info 2	Indicates the text written under Info 2
Info 3	Indicates the text written under Info 3
SOP	Indicates the name of the selected SOP
No entry	Indicates an empty field

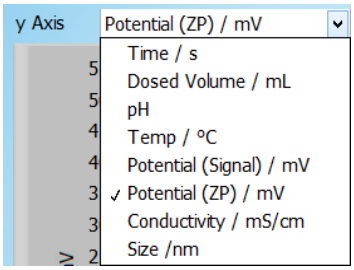
By selecting "Select Calibration" you can change from zeta potential to streaming potential and vice versa.

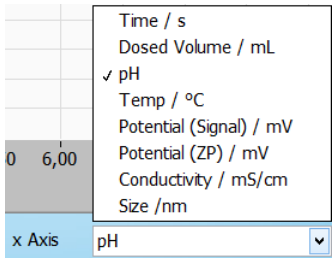
5.4. Graphic presentation of results

In a graphic presentation of measurement results you have a free choice for the denomination of the X- Y- parameter.



By clicking the drop-down menus at the Y- and X- axis (red arrows) you can select the desired parameter for the axis.
The following shows which presentation was selected: Measured Zeta potential vs pH.





Following choice is offered:

Time / s:	Duration of the measurement
Dosed Volume / mL:	Dosed volume of the titrand solution
pH:	Measured pH value
Temp / °C:	Measured temperature
Potential (Signal) / mV:	Measured streaming potential
Potential (ZP) / mV:	Measured zeta potential
Conductivity / mS cm^{-1}:	Measured conductivity
Size / nm:	Measured particle size. These data are only available if at begining of the measurement the option „Size Measurement“

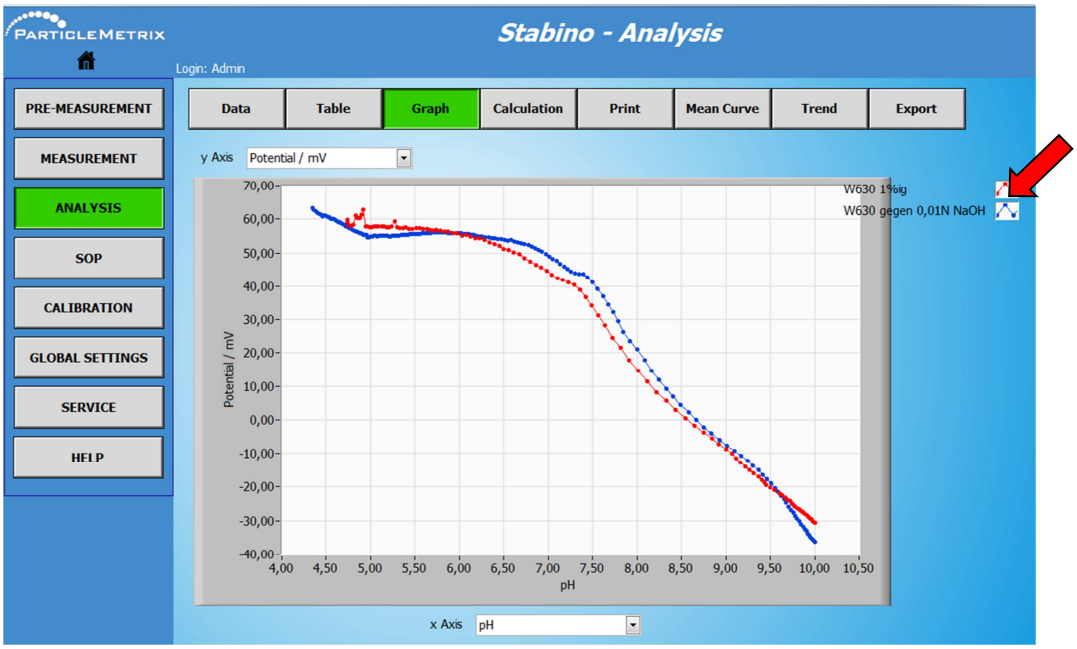
was activated and when the measured size was entered. The size measurement is not performed with the Stabino Control Software but with the Microtrac Flex Software.

To change the scaling of the graph you only have to click on the individual end and type in the new min. / max. value.

Graphic presentation after the scaling by yourself:

Note
When loading a new data file, the software performs an autoscaling again.

To change the style of the presentation by varying line width, line style, dashed or bolt, etc… click to the corresponding diagram symbol ( ) of the selected curve (red arrow).



Following dialogue window is opened:

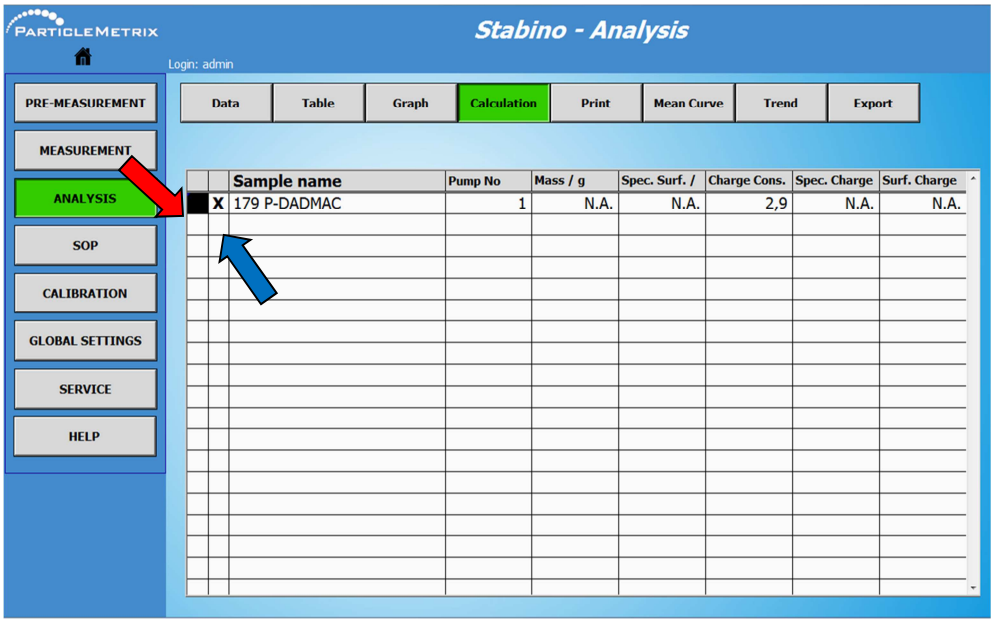
Here you can chose the desired color. The pre-selection of the colors one can define in the menu "GLOBAL SETTINGS" → Colors.

5.5. Calculations

Extended calculations are displayed under the menu „Calculation".

Note
These calculations are only possible with polyelectrolyte titrations and only when data of the sample and titrant solution were entered in the starting mask of „Calculation".

The structure of the table starts with the sample name in the first row (this is a fixed position and cannot be changed) .The next 6 columns can be set freely.



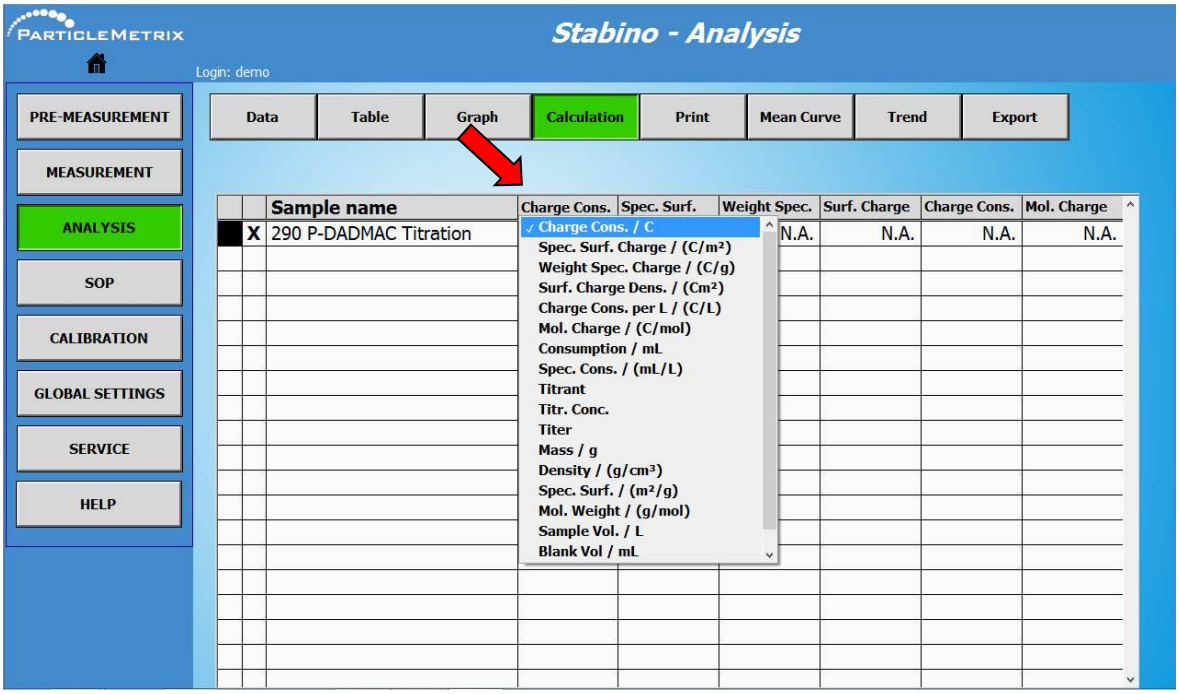
By clicking the "black" field (red arrow), the colour of the graph belonging to the sample name can be selected.

Following dialogue window opens:

Here you may select the desired color. The order of the colors can be preselected under the menu „GLOBAL SETTINGS" → Colors.

Should the selected sample be displayed as a graph or not, klick „X" (blue arrow).

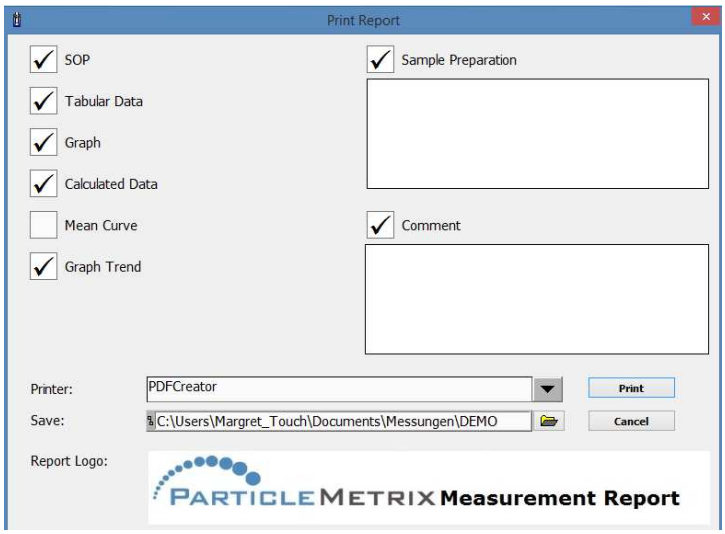
In each of the next 6 columns one of the listed measurement parameters or result data can can be chosen for being displayed in the record. To do this, click on top of the column (red arrow). The assortment menu opens with following selection possibilities.



Charge Cons. / C	Charge consumption to 0 mV *(Surface & Activity)*
Spec. Surf. Charge / C m^{-2}	Calculated specific surface charge *(Surface)*
Weight Spec.Charge / C g^{-1}	Calculated weight specific charge *(Surface & Activity)*
Surf. Charge Dens. / C m^{2}	Charge density on the surface
Charge Cons. per L / C L^{-1}	Charge consumption per Liter *(Activity)*
Mol.Charge / C mol^{-1}	Calculate the specific charge per Mol (*Activity)*
Consumption / mL	Consumption of titrand solution to titrate the sample to 0 mV (same value as V (0 mV) *(Surface & Activity))*
Spec.Cons. / mL L^{-1}	Specific consumption during the titration to 0 mV. The value is calculated from „Blank.Vol" and „Consumption" *(Activity)*
Titrant	Used titrand solution *(adopted from PRE-MEASUREMENT)*
Titr. Conc.	Concentration of the titrand solution *(adopted from PRE-MEASUREMENT)*
Titer	Titer of the titrand solution *(adopted from PRE-MEASUREMENT)*
Mass / g	Weighed portion of the sample *(Surface & Activity)*
Density / g cm^{-3}	Density of the sample *(Surface)*
Spec. Surf. / m^{2} g^{-1}	Specific surface of the sample *(Surface)*
Mol. Weight / g mol^{-1}	Molecular weight of the sample *(Acivity)*
Sample Vol. / L	Volume of the sample *(Activity)*
Blank. Vol / mL	Consumption of titrand solution to titrate the solvent to 0 mV *(Activity)*
No Entry	Indicates an empty field

5.6. Print data

To release the print of an analysis report click "Print".
Following windows opens.



By clicking the various check boxes you select which parameters / data should appear in a print. Following choice is offered:

SOP	SOP settings
Tabular Data	A table as defined under „Table"
Graph	A graph as set under „Graph"
Calculated Data	If in the start mode you entered further data for calculation, those data appear in the table as defined under „Calculation"
Graph Trend	The trend graph is printed as defined under „Trend"
Sample Preparation	The way how the sample was prepared should be entered here
Comment	Any further comments

By clicking on "Report Logo" you can change it.

Note
The logo must be offered in JPG format. Ideally it should have a size of 610 x 80 pixels.

In „Printer" you can select any printer, which is installed on your PC and print the report.

In the case you use a pdf – printer, under „Save" you can select where the pdf-document should be stored.

Note
All active data will be printed. The tables and graphs will be printed as selected and formatted before in the corresponding sections.

The measurement report has following appearance: It contains the date when it was created, the serial number of the Stabino and the software version used.

Stabino® Measurement Report

Stabino®
Charge Titration System

PARTICLEMETRIX Measurement Report

Created 2014-01-17 by Admin

Sample Preparation

SOP

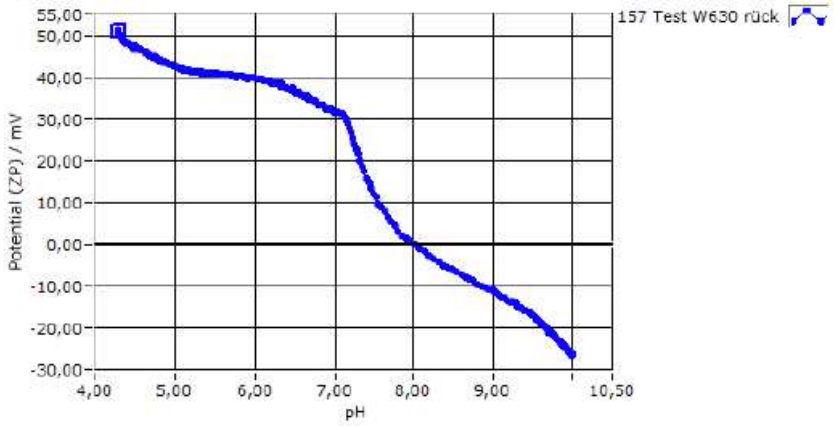
	Type	End Point	Pre-Dosage	Dosage Interval	Volume Increment
nach pH 10 fest 40 µL	pH	10,0 (pH)	N.A.	5,0 sec	40 µl

Measurement Data
Tabular Data
Select Calibration: ☐ Signal ☑ ZP

			Sample name	Titrant
1		X	157 Test W630 rück	NaOH 0,01000 N

		P (0 mL) / mV	V (0 mV) / mL	IEP	V (end pH) / mL	Piston
	X	51,2	7,287	8,0	10,617	400

Graph



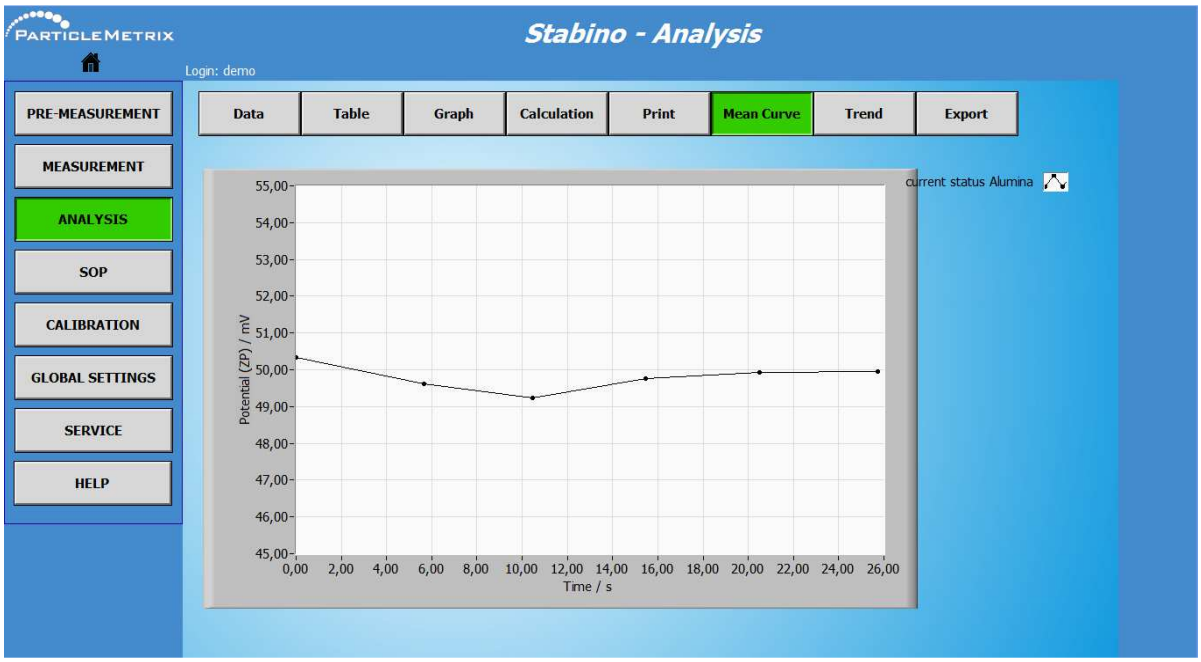
Comment

Stabino Control 2.00.13 Particle Metrix GmbH Stabino S/N:

5.7. Average values and averaged curves

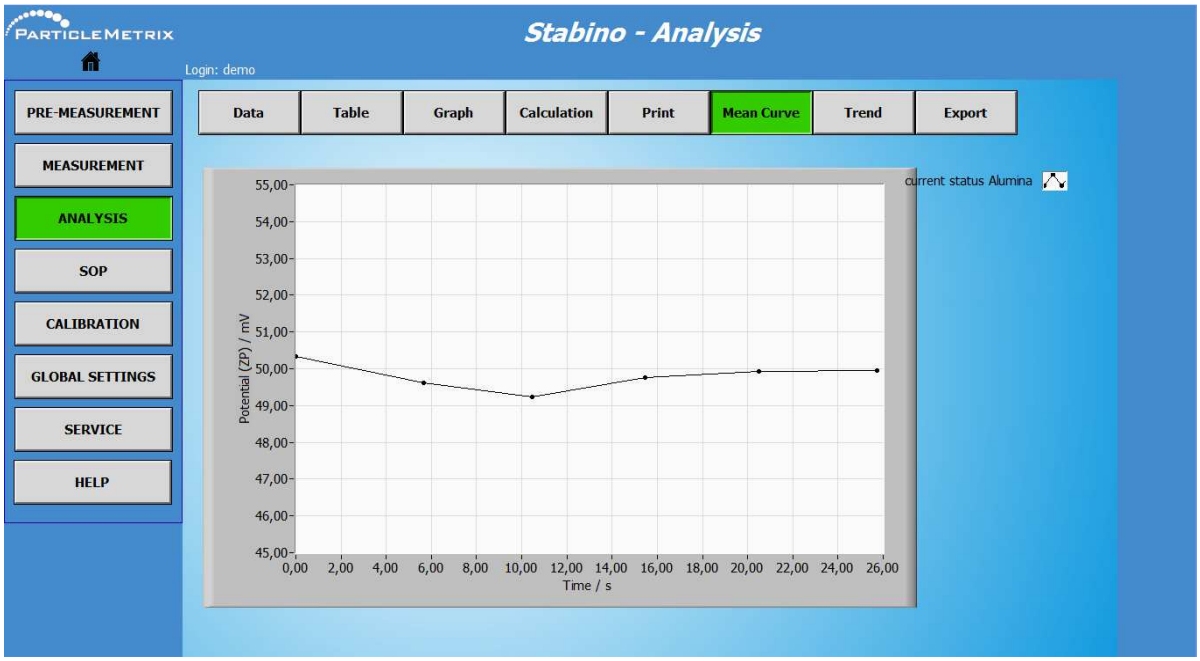
To show mean curves please choose “Mean Curve”. Here the mean curve is shown by all open measurements. The plot of the axis will show as choose under the "Graph". The scale bar can be changed as knowen from the graph.

Note
Mean curves can only be shown from idendical measurements with the same SOP and name. If there opend files with different SOP’s and samplenames the mean cuve will be shown from them with idendical behavior.



Overlay of three singel measurements:

Mean curve of the the single meausrements:

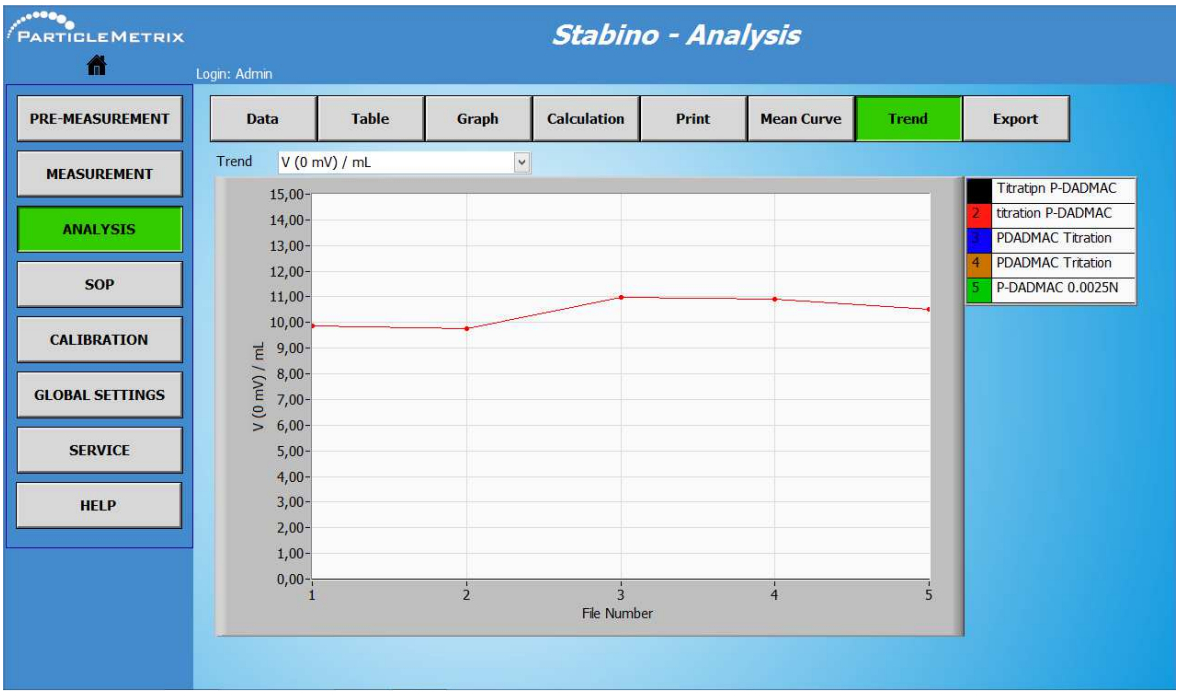


Attention!

In the case of mean curves of titration it is be possible the mean curve does not go through the zero point of charge or isoelectric point but the single measurements. This will be a mathematical problem.

5.8. Trend presentation

To show a trend of measurements click “Trend”.



		Sample name	Titrant	P (0 mL) /	V (0 mV) /	P max /	P min / mV	V (end) /
	X	275 Titratipn P-DADMAC	PVS 0,00250 N	63,6	9,879	64,2	-5,9	9,9
	X	264 titration P-DADMAC	PVS 0,00250 N	71,8	9,751	78,4	-5,3	9,8
	X	453 PDADMAC Titration	PVS 0,00250 N	65,2	10,976	65,3	-4,1	11,0
	X	284 PDADMAC Tritation	PVS 0,00250 N	63,3	10,921	66,5	-3,5	10,9
	X	14 P-DADMAC 0.0025N	PVS 0,00250 N	74,1	10,503	78,1	-0,2	10,5

Here you can display a trend of measurements in the sequence they were opened. Following parameters can be selected for a trend presentation:

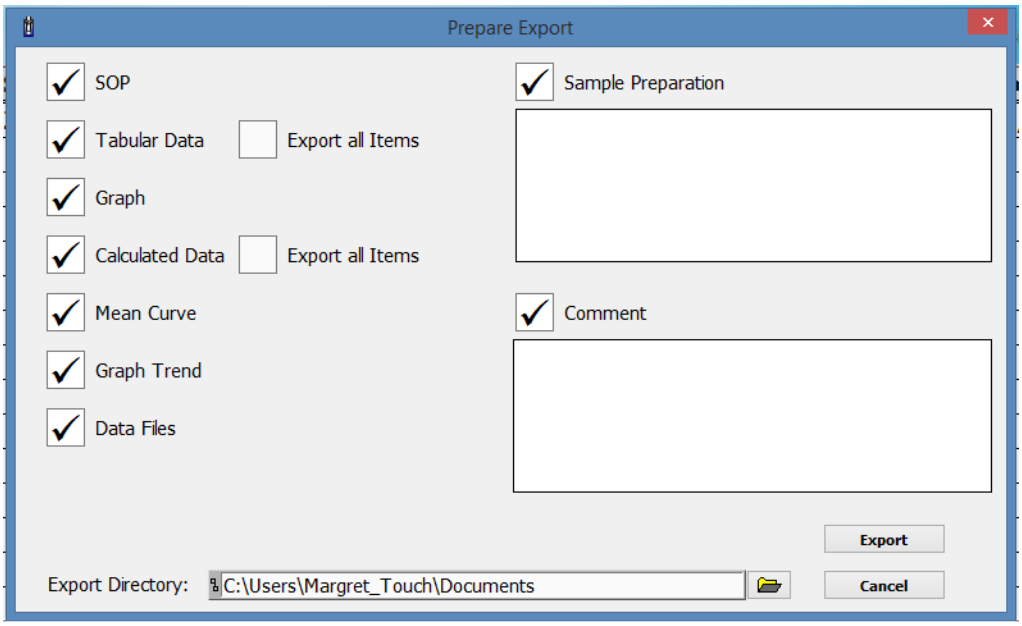
P (0 mL) / mV	Value of the starting potential
P max / mV	maximum potential
P min / mV	Minimum potential
P (end) / mV	Last measure potential
P (end pH) / mV	Measured potential at the end point pH
V (0 mV) /mL	Consumption of titrant solution to the zero point of charge
V (P max) / mL	Consumption of titrant solution to the maximum potential
V (P min) / mL	Consumption of titrant solution to the minimum potential
V (pH max) / mL	Consumption of titrant solution to the maximum pH value
V (pH min) / mL	Consumption of titrant solution to the minimum pH value
V (K max) / mL	Consumption of titrant solution to the maximum conductivity
V (K min) / mL	Consumption of titrant solution to the minimum conductivity
V (end) / mL	Total consumption of titrant solution
V (end pH) / mL	Consumption of titrant solution to the end-pH-value

pH (0 mL)	Value of the Start-pH
pH min	Maximum pH-value
pH max	Minimum pH-value
pH (P max)	pH-value at the maximum potential
pH (P min)	pH-value at the minimum potential
IEP	Isoelectric point
K (0 mL) / S cm^{-1}	Value of the conductivity at start
K (0 mV) / S cm^{-1}	Conductivity at the point of zero charge
K max / S cm^{-1}	Maximum conductivity
K min / S cm^{-1}	Minimum conductivity
K end / S cm^{-1}	Last measured conductivity
Size Type	Indicates the quantity (type) of the displayed size value
Mx max / nm	Maximum measured size
Mx min / nm	Minimum measured size
Mx (start) / nm	Size at starting point
Mx (end) / nm	Size at the end of the measurement
ABS (V (P min) – V (0 mV)) / mL	Consumption of titrant from the minimum to the zero point of charge
ABS (V (P max) – V (0 mV)) / mL	Consumption of titrant from the maximum to the zero point of charge
Date & Time	Date and time of measurement
Piston	Indicates which piston is used
Info 1	Indicates the text written under Info 1
Info 2	Indicates the text written under Info 2
Info 3	Indicates the text written under Info 3
SOP	Indicates the name of the selected SOP
No entry	Indicates an empty field

5.9 Export

To export measurement data click "Export".

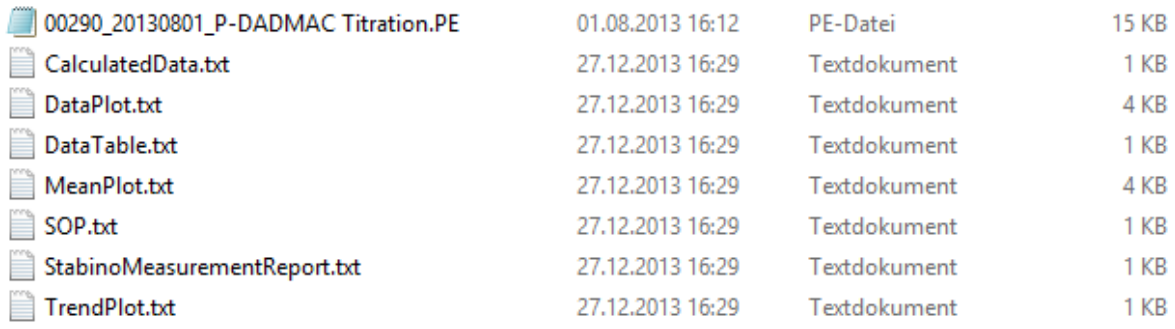
Following window is opened.



By clicking selected "Checkboxes" you choose which data are exported. The data are stored as TXT-Files. Following choices are available:

SOP	SOP settings
Tabular Data	A table as defined under "Table". With "**Export all items**" all data listed in a table are exported.
Graph	A graph as set under „Graph"
Calculated Data	If in the start mode you entered further data for calculation, those data appear in the table as defined under "Calculation". With "**Export all items**" all data listed in a table are exported.
Graph Trend	The trend graph is exported as defined under "Trend".
Sample Preparation	The way how the sample was prepared
Comment	Any further comments

Example of exported data:



00290_20130201_P-DADMAC Titration.PE	File name
CalculatedData.txt	Calculated data

DataPlot.txt	Graph
DataTable.txt	Tabular data
MeanPlot.txt	Average curves
SOP	SOP
StabinoMeasurementReport.txt	Sample preparation and comments
TrendPlot.txt	Trend plot

These data could be evaluated with various software.

6. Cleaning

6.1 Cleaning of the measurement cylinder and piston

To obtain reproducible and exact measurement results it is necessary to carefully clean the measurement cylinder and the piston before each measurement.

- Take out the measurement cell and empty it
- Rinse the measurement cell and the piston with water
- Clean the measurement cell and the piston carefully with the delivered brush.
- Before you continue, please read the safety instructions for using the cleaning solution given in chapter 8, or read the warning at the end of this page.
- In the case of having measured polyelectrolyte solutions it is recommended to clean the measurement cell and piston with the recommended cleaning solution.
- Fill 2-3 mL of cleaning solution (making up and using a cleaning solution see Chapter 8.) into the cell. Clean the measurement cell and the piston carefully with the delivered brush. **The cleaning action is not allowed as long as the measurement cell is attached to the instrument.**
- After the cleaning action, the measurement cell and piston have to be rinsed several times with deionized water.
- Dry the outside of the measurement cell with a clean tissue. Please dry the inside of the measurement cell and the piston by shaking drops away.

Notice
By no means, dry the inside of the measurement cell and the piston with a tissue. A tissue may contain additives which can give raise to an unwanted background measurement signal.

Notice
The measured sample must not rest in the measurement cell. The cell has to be cleaned as described. In the case the measurement cell is not used for a few days, the cell should be stored having it filled before with deionized water. In this way the drying of adsorbed impurities is avoided.
If the instrument is not used for more than a few days or in the case of transport, the measurement cell has to be emptied.

Notice
The cleaning solution should be selected corresponding to the sample.

Attention!
When preparing, storing and using solvents, the corresponding rules for hazardous products have to be followed. On the bottle containing the solution the constituents of the cleaning solution have to be clearly assigned.
The preparation of the cleaning solution is described in capture 8.1.

Security notice:
When you prepare or use the cleaning solution, carry protective glasses and wear corresponding to MSDS acetone. In particular, when cleaning the measurement cylinder and the piston with the brush, carry protective glasses to be protected against spilling solution.

6.2 Cleaning of the Stabino®

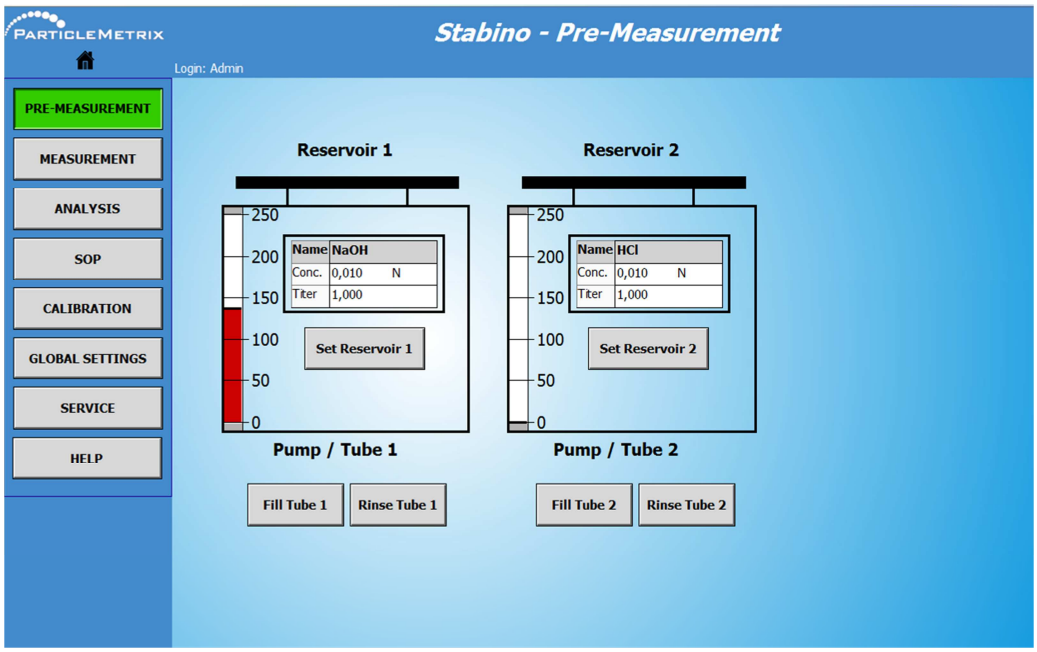
Please clean the outside of the Stabino® only with a soft wet tissue. The wetting liquid should be water with a mild cleaning agent added to it.

Attention!
By no means apply solvents or abrasive liquids. Applying them to clean the housing may ruin the paint and / or the writings on the housing.

Attention!
No liquid should be allowed to penetrate the inside of the housing. The liquid may eventually ruin electronics and mechanics.

6.3 Cleaning of the pump and tubing arrangement

To clean the hose and pump system of the Stabino® switch to the menu "Pre-Measurement" and click on button „Rinse Tube 1" or „Rinse Tube 2" depending on which pump-hose-combination you intend to clean.



Note
To avoid chemical reactions in the hoses or in the pump, clean the hose - pump system before changing the polyelectrolyte solution. Otherwise, the system may be spoiled.

Note
The cleaning procedure is parameterized in a way that it cannot be interrupted.

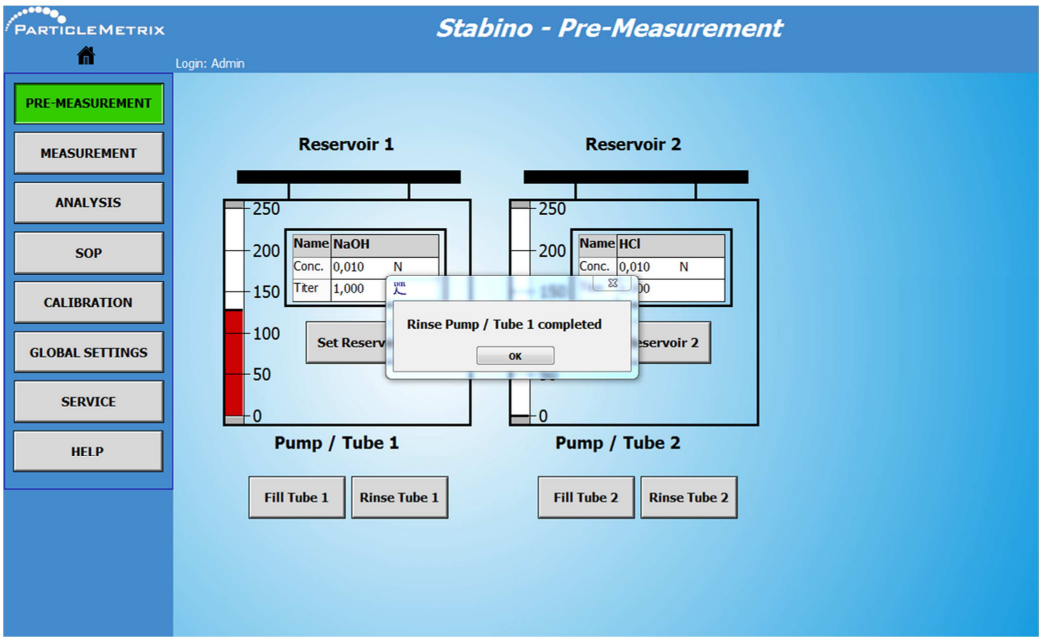
Subsequently follow the instructions of the monitor.

1
Dispose the titration solution

2
Rinse with water

3
Pump the hoses dry

Now the chosen hose / pump system is cleaned.



Attention!
If you do not use the system for a longer period, do not leave the hose / pump system filled with water or titration solution. Otherwise, you risk having deposits and biofilms in hoses and pumps.

7. Calibration of the Stabino®

For the calibration of the Stabino® click „Calibration" and the following window opens. In this menu the pumps, the pH reading, the signal and conductivity can be calibrated, the signal either as streaming potential or zeta potential. By clicking the corresponding buttons the desired calibration window opens.

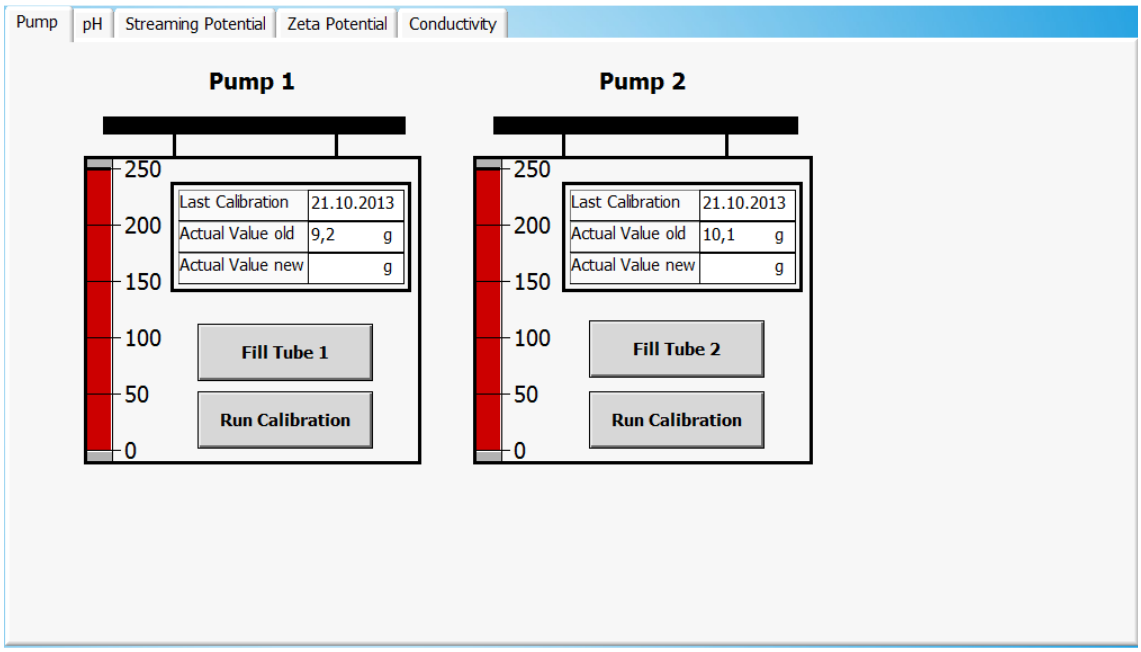
Attention

An exact measurement result can only be obtained, if the measurement system is carefully calibrated.

7.1 Pump-Calibration

Note: for the calibration of the pumps you need a precise balance and a clean beaker for a volume of approx. 15 ml.

By clicking the tab "Pump" the window for the pump calibration opens.
The calibration should be conducted with deionized or distilled water. Before you start the calibration, the hose / pump system must be filled with water. Tap to „Fill Tube". Subsequently you can start the calibration by „Run Calibration". Please follow the instructions on the screen. In the field "Last Calibration" the date of the last calibration action can be seen.



Note A fixed number of pump strikes is performed. From the weighted masses the pump volume per strike is activated.

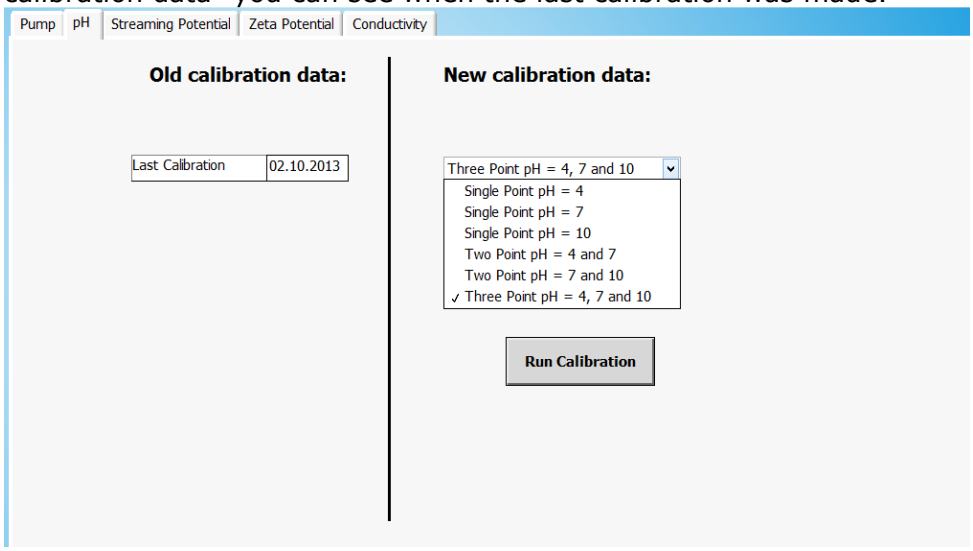
400 strikes of the pump are always applied by default. The pump calibration in µL/strike is deducted from the measured weight divided by 400 strikes.

7.2 pH Calibration

By selecting the "pH" tab, you open the pH calibration procedure. Various kinds of calibration can be selected:

Single Point pH = 4:	The calibration is performed only on pH = 4
Single Point pH = 7:	The calibration is performed only on pH = 7
Single Point pH = 10:	The calibration is performed only on pH = 10
Two Point pH = 4 and 7:	The calibration is performed on pH = 4 and 7
Two Point pH = 7 and 10:	The calibration is performed on pH = 7 and 10
Three Point pH = 4,7 and 10:	The calibration is performed on pH = 4, 7 and 10

To start the pH calibration, select the desired type and tap on "Run Calibration". Subsequently you have to follow the instructions given on the screen. In the field "Old calibration data" you can see when the last calibration was made.



Note

The protection cap of the pH sensor should be filled with a 3 molar KCl Potassium Chloride solution. This salt solution is necessary to avoid desiccating of the diaphragm of the pH probe. It keeps the electrode at the correct salt solution and eliminates an eventual diffusion pressure.

Attention!

By no means should the pH probe be stored in distilled water! Otherwise KCl diffuses through the diaphragm to the outside. The reference electrolyte concentration decreases. As a consequence, the pH probe will be deteriorated irreversibly.

Note

Basic buffer solutions should be stored in a closed and calibrated vessel. In the presence of air CO2 may change the pH value.

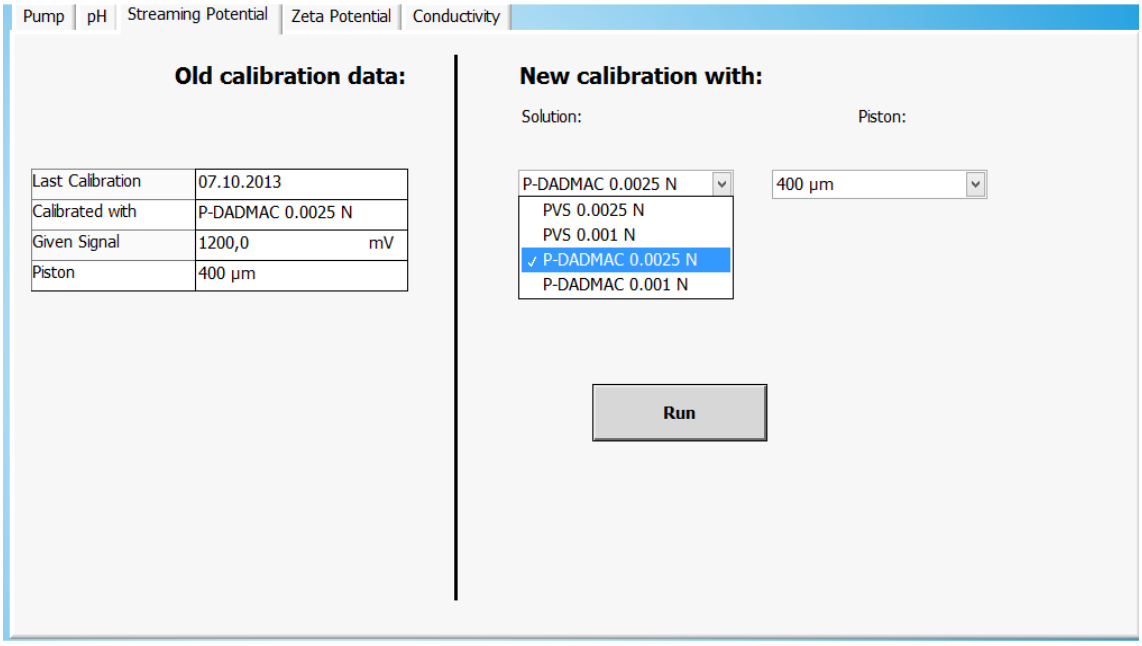
Note

Contamination of the buffer solutions have to be avoided. Therefore it is recommended to never dip the pH electrode into the reservoir of the buffer solution. Extract just the quantity from the container, which is needed. Close up the reservoir bottle immediately after use!

7.3 Calibration of the signal

By tapping on "Signal", the menu Signal-Calibration is opened. Here it is possible to calibrate the signal to the well-established streaming potential known from StabiSizer®.

To start the calibration it is necessary to select the solution, with which the calibration is intended to be performed. Solution of 0.001 N or 0.025 N PVS (-1200 mV) or 0.001 N and 0.025 N P-DADMAC (+1200 mV) respectively can be used. After having chosen the kind of solution, the piston has to be selected, which is taken for the calibration. It is recommended to perform a calibration with P-DADMAC and the 400 µm piston. Start the calibration with "Run" and follow the instructions on the screen.
In the field "Last Calibration" the date of the last calibration action can be seen.



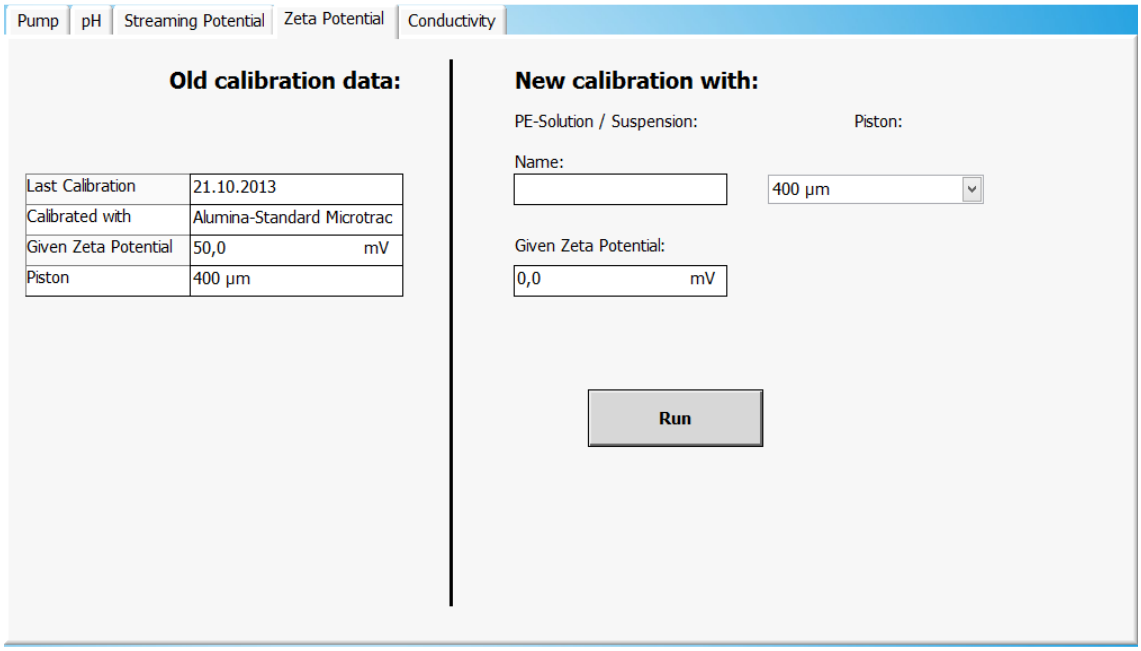
Note
Different pistons lead to different potential signals of the same signal. The signal depends on the depth of the grove in the piston. The depth is responsible for the fluid velocity in the gap between the cylinder and the piston. The speed is responsible for the sheer force of the liquid on the particles which are adsorbed to the Teflon wall. **The signal should be calibrated with a 0.001 N Poly-DADMAC solution and a 400 µm piston.** With his calibration a signal of +1200 mV is generated. Should the signal change by ±4%, it is recommended to clean the measurement cylinder and the piston.

7.4 Calibration of the signal as zeta potential

By tapping on „Charge / Zeta Potential", the menu Signal-Calibration for a zeta potential reading is opened. Following these instructions the calibration of the signal as zeta potential is performed.

Before starting the calibration, write the name of the standard zeta potential suspension into the field „Name" and enter the value of the zeta potential into the box „Given Zeta Potential". The value is given on the certificate of the standard. Hereafter the type of piston has to be entered, with which the zeta potential calibration is performed. Now start the calibration with „Run" and follow the instructions on the screen.

Under „Old Calibration Data" you see, when and how the last calibrations were made.



Note

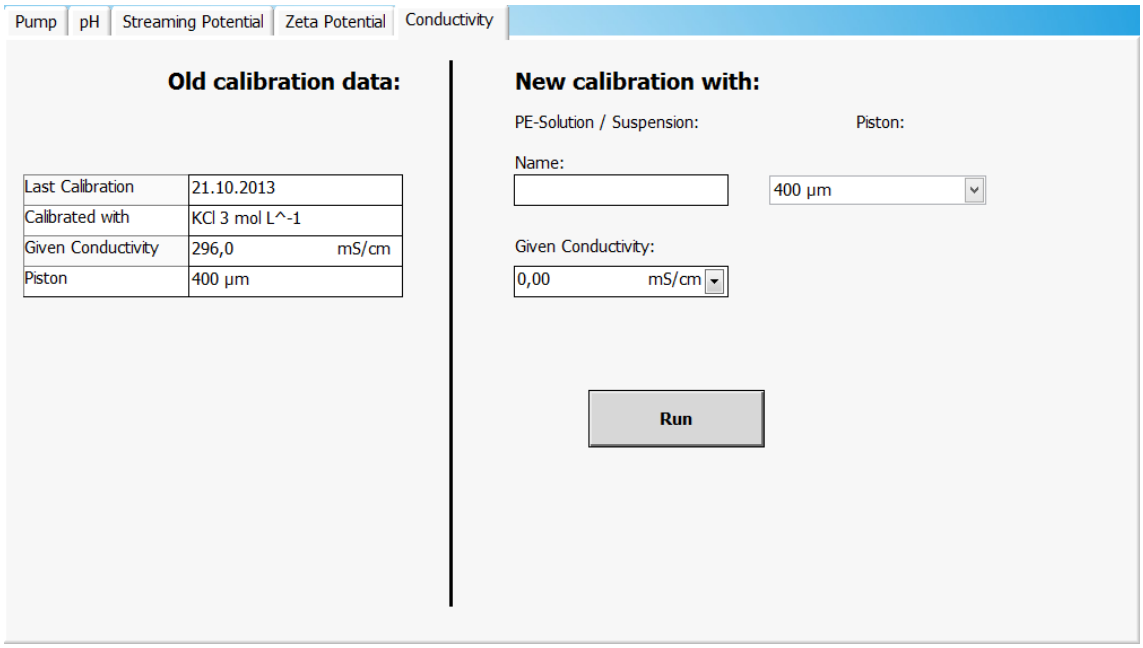
Different pistons cause different signals of the identic sample. The signal depends of the groove depth of the piston, as this determines the speed of the fluid.

7.5 Calibration of Conductivity

With a click on "Conductivity" you reach the conductivity calibration. Here, the operator performs the conductivity measurement. For starting the calibration you type in the name of the calibration standard into the field „Name". Into the field "Given Conductivity" you type in the real conductivity of the standard. With the drop down menu the entity in mS/cm or µS/cm can be selected. (The µ is displayed as u).

After entering the data you must choose the piston for calibration. To start a calibration, click on "Run" and follow the display instructions.

Under "Old calibration data" you see the data of the last calibration.



Note
You can use different conductivity standards. The most used standard is a 0,01 N KCl-solution with a conductivity of 1,41 mS cm^{-1} .

8. Annex

8.1 Making up a cleaning solution

Use following solution as a universal cleaning solution for the manual cleaning of the measurement cell and the piston:

- 70 parts of deionized water
- 30 parts of acetone

Attention!
When preparing, storing and using solvents, the corresponding rules for hazardous products have to be followed.
The constituents of the cleaning solution have to be clearly assigned on the bottle containing the solution.

Security notice:
When you prepare or use the cleaning solution, carry protective glasses and wear corresponding to MSDS acetone. In particular, when cleaning the measurement cylinder and the piston with the brush, carry protective glasses.

Security advice
Acetone is easy flammable and must be separated from heating points and open fire. The opening of closed bottles with acetone or acetone-water mixture has to be handled carefully, because the bottles could have an overpressure. These bottles have to be stored under cooling. Contact with the skin: Please swill efficiently with water. Contact with eyes: Spill with open lid multiple minutes with water.

Preparing the solution

Acetone evaporates easily; therefore you have to prepare the cleaning solution under a special outlet. As a common cleaning solution for manual cleaning of cell and piston please use following mixture:

- 70% de-ionized water
- 30 % Acetone

The water has to be used first.

8.2 Maintenance

The Stabino is more or less maintenance free. The duties to be conducted by the user to keep the instrument producing good results narrow down to

- Cleaning of the measurement cell and piston (see chapter 6.1)
- Cleaning of parts of the housing (see chapter 6.2)
- Cleaning of the pump system (see chapter 6.3)
- Calibration of the pumps (see chapter 7.1)
- Checking the signal of the pH electrode (see chapter 7.2)
- Checking the signal of the piston in use (see chapter 7.3)

Safety warning:

Electric Power!
During cleaning the housing with a moist stuff, the instrument must be switched off and unplugged from the electrical supply. The instrument should not be cleaned with spray or with over wetted stuff. Do not use dash bottles.

Personal protective equipment
When using chemicals for the cleaning of the measurement cell or for the pistons, safety measures corresponding to the expected danger have to be applied. In particular, it is obligatory to carry protective glasses when using the brush. The safety data sheets (MSDS) of the liquids have to be carefully read, the instructions to be obeyed.

8.3 Spare parts

The spare parts which can be replaced by the customer are obtainable from Particle Metrix GmbH / Microtrac Europe GmbH or from the local distributor.

Piston 100 µm
Piston 200 µm
Piston 400 µm
Piston 1.000 µm
Measurement cylinder

- Cover for measuring cylinder
- pH electrode
- Calibration dispersion "Zetapotential"
- Calibration dispersion "Streaming potential, cationic" *)
- Calibration dispersion " Streaming potential, anionic" *)
- Cleaning brush for the measurement cylinder
- set of PTFE tubes
- Buffer pH=4 *)
- Puffer pH=7 *)
- Puffer pH=10 *)
- Cleaning brush
- Tubing set PTFE

***) In order to avoid shipping of chemicals, it is recommended to find a local supplier**

8.4 Troubleshooting

Trouble	Cause	Countermeasure
The power on LED is not on	On/Off-switch in „O"-Position	Bring On/Off-switch in Position „I"
	Power plug disconnected	Connect power plug
	Power adaptor defect	Exchange against an adaptor of original type
	Fuse defect	Exchange fuse (see chapter 8.5)
pH-signal cannot be calibrated	Buffer solution too old	Use new buffer solution
	pH electrode outdated	Use new pH electrode
Streaming potential / zeta-potential showing wrong value of the calibration solution / dispersion (before calibration)	Measurement beaker / piston dirty	Clean measurement beaker / piston
Streaming potential / zeta-potential showing wrong value at calibration solution / dispersion (after calibration)		Call service of Particle Metrix or contracted service organization
Piston hits the bottom of the measurement cylindER (regular noise at 5Hz)	Upper FIXATION is in the wrong position	Call service of Particle Metrix or contracted service organization

In other cases please call the service of Particle Metrix or the contracted service organization.

8.5 Exchanging the fuse

Warning:

Please respect the information on the stickers of the power adaptor to find the correct fuses, which correspond to the type of instrument.

The exchangeable fuse is located inside the power cable socket of the power adaptor. Please disconnect the cable from the power line and from the power adaptor. Please use only original replacement fuses, glass fuse 230V, 0.400 A (slow) and glass fuse 115V, 0.800 A (slow) for power adaptors to 230 Volt and to 115 Volt respectively.

Note: Picture just for showing position of fuses. **Rating may be different, according to instruments**.

Procedure:
1. Switch the main switch of the Stabino to „O" (OFF).
2. Pull the connection cable from the mains socket.
3. Pull the connection cable from the power adaptor.
4. By taking a small screw diver, the capping of the fuse can be pushed in and lifted:

5. Pull out the shelf, which is holding the fuse. Replace the defect fuse with a functioning one.

6. Slide the shelf in again, until it snaps in.
7. Plug the connection cable to the power adaptor.
8. Plug the connection cable to the mains socket.
9. Switch the mains switch of the Stabino to „I" (ON).

In the case that the trouble remains, please contact the technical service of Particle Metrix GmbH or call service of Particle Metrix or the contracted service organization.

8.6 Disposal

After expiration of the life time of the Stabino, the instrument has to be orderly disposed respecting the rules of the local authorities.
The disposal of the used chemicals has to be accomplished by following the corresponding regulations.