Measure what is measurable and make measurable that which is not.

Galileo Galilei (1564-1642)
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Instruction Manual and Safety Information

SurPASS 3
SurPASS 3 Eco

(Original Instruction)
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1 Safety Instructions

- Read the documentation before using the SurPASS 3 instrument.
- Follow all hints and instructions contained in the documentation to ensure the correct use and safe functioning of the SurPASS 3 instrument.
- The documentation is a part of the product. Keep this document for the complete working life of the product and make sure it is easily accessible to all people involved with the product. If you receive any additions or revisions to the documentation from Anton Paar GmbH, these must be treated as part of the documentation.

1.1 General Safety Instructions

Liability
- The documentation does not claim to address all safety issues associated with the use of the instrument and samples. It is your responsibility to establish health and safety practices and determine the applicability of regulatory limitations.
- Anton Paar GmbH only warrants the proper functioning of SurPASS 3 if no adjustments have been made to the mechanics, electronics, and firmware. Do not perform any modifications on the SurPASS 3 instrument.
- Only use SurPASS 3 for the purpose described in the documentation. Anton Paar GmbH is not liable for damages caused by incorrect use of SurPASS 3.

Installation and use
- SurPASS 3 is not an explosion-proof instrument and therefore must not be operated in areas with risk of explosion.
- The installation procedure should only be carried out by authorized personnel who are familiar with the installation instructions.
- Do not use any accessories or spare parts other than those supplied or approved by Anton Paar GmbH.
- Make sure all operators are trained to use the instrument safely and correctly before starting any applicable operations.
- In case of damage or malfunction, do not continue operating SurPASS 3. Do not operate the instrument under conditions which could result in damage to goods and/or injuries and loss of life.
- If you observe leakage underneath the SurPASS 3 instrument, immediately stop the measurement and disconnect the SurPASS 3 instrument from its power supply. Identify the reason for leakage and contact your Anton Paar representative.
- Check SurPASS 3 for chemical resistance to the samples and cleaning agents. See section 6.5 Wetted Parts for a list of materials that come in contact with the electrolyte solution and titration liquids.

Maintenance and service
- The results delivered by SurPASS 3 not only depend on the correct functioning of the instrument, but also on various other factors. We therefore recommend you have the results checked (e.g. plausibility tested) by skilled personnel before consequential actions are taken based on the results.
- Service and repair procedures may only be carried out by authorized personnel or by Anton Paar GmbH.

Disposal
- Concerning the disposal of SurPASS 3, observe the legal requirements in your country.

Returns
- For repairs send the cleaned SurPASS 3 to your Anton Paar representative. Only return the instrument together with the filled out RMA (Return Material Authorization) and the form "Safety Declaration for Instrument Repairs". Please download the Safety Declaration form from our website www.anton-paar.com.
- Do not return instruments which are contaminated by radioactive materials, infectious agents or other harmful substances that cause health hazards.

Precautions for highly inflammable samples and cleaning agents
- Observe and adhere to your national safety regulations for handling the measured samples
1 Safety Instructions

(e.g. use of safety goggles, gloves, respiratory protection etc.).

- Only store the minimum required amount of sample, cleaning agents and other inflammable materials near SurPASS 3.
- Do not use inflammable, toxic or explosive samples with SurPASS 3.
- Do not spill sample/cleaning agents or leave their containers uncovered. Immediately remove spilled sample/cleaning agents.
- Make sure that the setup location is sufficiently ventilated. The environment of SurPASS 3 must be kept free of inflammable gases and vapors.
- Connect SurPASS 3 to the mains via a safety switch located at a safe distance from the instrument. In an emergency, turn off the power using this switch instead of the power switch on SurPASS 3.
- Supply a fire extinguisher.
- Ensure the sufficient supervision of SurPASS 3 during operation.

1.2 Conventions for Safety Messages

The following conventions for safety messages are used in this instruction manual:

**WARNING**

*Description of risk.*
Warning indicates a hazardous situation which, if not avoided, could result in death or serious injury.

**CAUTION**

*Description of risk.*
Caution indicates a hazardous situation which, if not avoided, could result in minor or moderate injury.

**NOTICE**

*Description of risk.*
Notice indicates a situation which, if not avoided, could result in damage to property.

1.3 Safety Signs on the Instrument

![Fig. 1: Warning signs on the instrument where the measuring cells are to be connected](image-url)

**CAUTION**

*Electrostatic sensitive device*
Caution indicates a situation which, if not avoided, could result in damage to property.

- **Wear protective gloves**
  Wear protective gloves when handling the SurPASS 3 instrument.

- **Wear safety goggles**
  Wear safety goggles when handling the SurPASS 3 instrument.

- **Use protective clothing**
  Use protective clothing when handling the SurPASS 3 instrument.

**TIP:** *Tip gives extra information about the situation at hand.*

**WARNING**

*Hazardous liquids*
The electrolyte circuit may contain hazardous liquids. Make sure to empty the electrolyte circuit before removing the measuring cell.
**CAUTION**

*Electrostatic sensitive device*

SurPASS 3 is an ESD sensitive device. Do not touch the electrodes.

**WARNING**

*Hazardous liquids*

The titration unit of SurPASS 3 is intended for use with acid and base. Always empty the titration unit and rinse it before removing the titration hoses.

**NOTICE**

Take care that the warning symbols remain clearly legible.

---

*Fig. 2: Warning sign on the SurPASS 3 titration unit (not applicable to SurPASS 3 Eco)*
Thank you for buying SurPASS 3 for the zeta potential analysis of macroscopic solid surfaces.

Fig. 3: SurPASS 3 instrument

The SurPASS 3 instrument is available in two different configurations: As fully equipped SurPASS 3 for high-end zeta potential analysis as it is described in this instruction manual, and as entry-level device for single-point measurements, SurPASS 3 Eco. With SurPASS 3 Eco the recording of automated pH dependences and studies of adsorption kinetics are disabled. Please note that SurPASS 3 Eco is upgradeable to a fully equipped SurPASS 3 on-site by an Anton Paar certified service engineer, in case you wish to exploit the full potential of your SurPASS 3 instrument in the near future.

TIP: In this instruction manual, instrument and software features which are not accessible with SurPASS 3 Eco, are labeled as such.

Together with the SurPASS 3 instrument you have received the SurPASS 3 software for the remote control of SurPASS 3 with a PC or laptop computer.

The SurPASS 3 software offers the following functions:

- **Remote control of the SurPASS 3 instrument**
  The used measuring cell is automatically detected and the zeta potential analysis can be started at the click of a button. All key parameters are displayed in a single view.

- **Automatic detection of the isoelectric point (not applicable to SurPASS 3 Eco)**
  SurPASS 3 detects whether acidic or alkaline titration has to be applied for the automated determination of the isoelectric point of a sample.

- **Automatic pH scans (not applicable to SurPASS 3 Eco)**
  Complete pH titrations can be performed fully automatically over a desired pH range.

- **Adsorption kinetics (not applicable to SurPASS 3 Eco)**
  Time-resolved adsorption studies from the liquid phase onto solid surfaces can be performed by monitoring the change in streaming potential or streaming current while changing the composition of the electrolyte solution.

- **Display and export of data**
  Measurement data can be loaded and displayed at any time. It is also possible to compare measurement results of different files. Furthermore, all data are automatically saved for user-specific evaluation in a spreadsheet software.

### 2.1 Intended Use of the Instrument

The SurPASS 3 instrument is applicable for the zeta potential analysis of solids of almost any shape and size like fibers, foils, sheets, textiles, powders or granules. The principle for the zeta potential analysis with the SurPASS 3 instrument is based on the streaming potential and streaming current method. In combination with the integrated titration unit, it is possible to investigate the dependence of the zeta potential on the pH fully automatically, thus determining the isoelectric point of a sample surface.

The SurPASS 3 instrument is intended for use with dilute aqueous solutions of inorganic salts, acids and bases. For standard measurements, the background electrolyte solution composes of a 0.001 mol/l aqueous NaCl or KCl solution. For adjusting the pH value of the background electrolyte solution, the usage of 0.05 mol/l HCl and 0.05 mol/l NaOH or KOH, respectively, is recommended.
2.2 Functional Elements

2.2.1 Front View

Fig. 4: SurPASS 3 front view. The green LED (1) indicates that the instrument is switched on.

2.2.2 Rear View

Fig. 5: SurPASS 3 Eco rear view
1 power switch
2 connector for pH electrode
3 connector for conductivity electrode
4 connector for USB cable

2.2.3 Right Side View

Fig. 6: SurPASS 3 rear view
1 power switch
2 connector for pH electrode
3 connector for conductivity electrode
4 connector for USB cable
5 connector for CAN cable for pressure control unit

Fig. 7: Right side view of SurPASS 3 Eco
1 3-way valve of SurPASS 3 titration unit
2 1.0 ml syringe of SurPASS 3 titration unit
3 piston sled of SurPASS 3 titration unit
2.3 System Requirements

The following hardware configuration is required to successfully install and run the SurPASS 3 software on your computer:

- Intel® Core™ i3 CPU
- 4 GB RAM (depends on the operating system)
- Screen resolution of at least 1024 x 768 at True Color (32 bit), best experience with a monitor with an aspect ratio of 16:9 or 16:10
- Use of a dedicated graphics card that supports Direct X 9 is recommended
- 20 GB free disk space on system drive
- Mouse and keyboard

The SurPASS 3 software can be installed on one of the following operating systems:

- Microsoft Windows® 7
  - 32 bit and 64 bit are supported
  - Service Pack 1 or later is required
  - Windows 7 Starter Edition is not supported
- Microsoft Windows® 8
  - 32 bit and 64 bit are supported
- Microsoft Windows® 10
  - 32 bit and 64 bit are supported

* Microsoft Windows® Server 2008 / 2008 R2
  - 32 bit and 64 bit are supported
  - Service Pack 2 or higher is required for Windows Server 2008

* Microsoft Windows® 10
  - 32 bit and 64 bit are supported

NOTICE
Please make sure that all requirements are fulfilled to guarantee trouble-free operation of the SurPASS 3 software.

2.4 Water Quality and Measurement Conditions

The zeta potential is very sensitive to the outermost solid surface. Care needs to be taken when it comes to the selection of the chemicals used for measurement. High purity chemicals need to be used for measurement, since contamination may affect the analyses especially of hydrophobic samples or samples with a small surface area (e.g. polymer films).

<table>
<thead>
<tr>
<th>Table 1: Required chemicals for successful SurPASS 3 measurements</th>
</tr>
</thead>
<tbody>
<tr>
<td>ultrapure water (ASTM I) for rinsing the electrolyte hoses and for preparing the electrolyte solution for measurement</td>
</tr>
<tr>
<td>KCl dry salt, &gt; 99.5 % p.a. or NaCl dry salt, &gt; 99.5 % p.a. for the preparation of 0.001 mol/l electrolyte solution</td>
</tr>
<tr>
<td>0.05 mol/l HCl* acid for pH titration</td>
</tr>
<tr>
<td>0.05 mol/l NaOH or KOH* base for pH titration</td>
</tr>
<tr>
<td>three buffer solutions (e.g. pH 3, 7, 10) for pH calibration</td>
</tr>
<tr>
<td>0.1 mol/l KCl solution or conductivity standard for conductivity calibration</td>
</tr>
</tbody>
</table>

* A concentration of 0.05 mol/l is best suited for the pH titration in the range of pH 3-9. If the zeta potential analysis is required at lower or higher pH, the concentration of the stock solution of acid and base must be increased accordingly.
A crucial requirement for a successful measurement is the quality of water, which is used for the operation of the SurPASS 3 instrument. The water used must comply with the American Standard for Testing and Materials I (ASTM I).

### Table 2: Minimum ASTM I standard

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Requirement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conductivity (at 25 °C)</td>
<td>0.056 µS/cm</td>
</tr>
<tr>
<td>Electric resistivity (at 25 °C)</td>
<td>18.0 MΩ cm</td>
</tr>
<tr>
<td>TOC</td>
<td>max. 100 µg/l</td>
</tr>
<tr>
<td>pH value*</td>
<td>5.5 - 6.5</td>
</tr>
<tr>
<td>* not specified by ASTM I</td>
<td></td>
</tr>
</tbody>
</table>

**NOTICE**

- It is not possible to perform successful SurPASS 3 verification measurements (see chapter 4.4) if the water quality is not sufficient. In the worst case, the water used for measurement can even seriously contaminate the electrolyte circuit.
- Commercial water purification units are specified in terms of ASTM standards but only provide the indicated water quality if they are also maintained regularly, in intervals as recommended by the manufacturer.
- The use of bottled ultrapure water is possible if no appropriate water purification unit is available.
- It is recommended to continuously purge the electrolyte solution with nitrogen 5.0 throughout the duration of the measurements. Do not use compressed air or inert gas with a lower quality.

NaCl or KCl used for the fresh preparation of the measuring electrolyte needs to be *pro analysis* grade. It is important that the salt is dry and not contaminated by any other chemical. It is recommended to use fresh NaCl *p.a.* or KCl *p.a.* for preparing the measuring electrolyte solution by putting 15 mg of KCl salt (12 mg of NaCl) and dissolving them in 200 ml ultrapure water. The preparation of stock solutions is not recommended. It is not recommended to use ready-made NaCl or KCl solutions, as they may contain stabilizers which also affect zeta potential measurements.

Acid and base come in contact with the electrolyte system and the sample during pH titration. It is recommended to exchange the 0.05 mol/l HCl and 0.05 mol/l NaOH (or KOH) solutions after every month.

**WARNING**

**Risk of injury**

Acids and bases may only be handled by skilled personnel who are aware of all risks associated to the used liquids. Refer to the reference guide D85IB016EN for the safety data sheets of 0.1 mol/l HCl and 0.1 mol/l NaOH.

- **Wear protective gloves**
  Wear protective gloves when handling acids and bases.
- **Wear safety goggles**
  Wear safety goggles when handling acids and bases.
- **Use protective clothing**
  Use protective clothing when handling acids and bases.
3 Preparing the Instrument

3.1 Placing the SurPASS 3

SurPASS 3 is a benchtop instrument. Select a suitable place for your SurPASS 3 instrument to avoid incorrect measurements.

- Make sure that the SurPASS 3 is placed on a table in a horizontal position to avoid an overflow of electrolyte from the collecting dike in case of malfunction.
- Make sure that the SurPASS 3 is placed in a way to reach the power switch easily.
- Do not place SurPASS 3 near heat sources.
- Avoid direct exposure of SurPASS 3 to sunlight.
- Prevent influence by vibrations.

**TIP:** When moving SurPASS 3, lift the instrument at the front and rear bottom.

3.2 Removing the Transport Lock

**NOTICE**

Before putting the SurPASS 3 instrument in operation, you need to connect the cable of the hydraulic valve motor and remove the transport lock (Kapton foil) that protects the hydraulic valve.

If you fail to do so before starting a process (e.g. Rinse), the SurPASS 3 software will display an error message:

**Rinse failed**

```
The operation failed on SurPASS 3
Error code: 02100008h
Please contact customer support and provide the error details.
Operation type: 4 'Current' sequence
Operation error code: 1 'Not translated'
Operation status code: 8 'Sequence failed'
```

Instructions for removing the transport lock:

1. Make sure the SurPASS 3 instrument is switched off.
2. Remove the two knurled screws in the corners of the front plate.
3. Pull out the plug-in module completely.

![Fig. 9: Front plate of the plug-in module](image)

4. Take the plug of the hydraulic valve motor from its support in the Kapton foil.

![Fig. 10: Plug-in module](image)

5. Connect the plug of the hydraulic valve motor to the connector on the back housing.

![Fig. 11: Removing the Kapton foil from the plug](image)
3 Preparing the Instrument

6. Insert the blade of the T20 Torx T-handle screw driver into the corresponding holes on the back side of the plug-in module housing. Support the valve motor and open the two screws to a small extent, such that the Kapton foil gets movable.

7. Pull out the Kapton foil that protects the hydraulic valve. Make sure the Kapton foil is pulled out as a whole. Fix the two screws again, hand-tight only.

8. Insert the plug-in module into the SurPASS 3 housing.

9. Lock the front panel by fixing the knurled screws on the bottom of the front panel.

**NOTICE**
The Kapton foil must NOT be discarded. If the instrument is kept out of operation for an extended period, the Kapton foil must again be placed between the ceramic discs - refer to section 9.7.4 in the reference guide D85IB016EN.

### 3.3 Mains Connection

The power cable receptacle and the power switch are located at the rear of the SurPASS 3 instrument (pos. 1 in Fig. 6). The mains voltage is indicated at the back side of SurPASS 3.

#### CAUTION

**Mains Voltage**
SurPASS 3 is designed for operation on mains fulfilling the following requirements:
115/230 VAC, 50/60 Hz, ± 10 % allowable mains voltage tolerance.

#### WARNING

**Risk of injury**
If you notice that the power cable is defective, immediately disconnect SurPASS 3 from the power supply. Replace the power cable.

#### 3.3.1 Protective Grounding

Grounding of the SurPASS 3 instrument is done through a three-wire power cable which has to be connected to a protective ground connector socket.

No other kind of grounding is permitted.
3 Preparing the Instrument

3.4 PC Connection

The SurPASS 3 instrument is controlled by a PC with the Microsoft Windows® based SurPASS 3 software. SurPASS 3 communicates with the PC via a USB cable which is connected at the rear side of SurPASS 3 in the socket USB (pos. 4 in Fig. 6).

3.5 Mounting the Electrolyte Hoses

The electrolyte hoses inside the SurPASS 3 instrument have already been mounted before shipment.

1. Pull the loose ends of the two hoses through the corresponding holes of the white beaker cover.

3.6 Connecting the Conductivity and pH Electrodes

The conductivity electrode is equipped with a temperature sensor and a single connector cable.

1. Plug the conductivity electrode cable in the socket C/T of the connector panel on the backside of the SurPASS 3 instrument (pos. 3 in Fig. 6).

2. Place the conductivity electrode in an appropriate hole of the beaker cover (pos. 2 in Fig. 15).

![Fig. 15: Drawing of the beaker cover](image)

1 Position for pH probe
2 Position for conductivity electrode
3 Positions (for acid and base dispense) for titration hoses
4 Position for nitrogen purge hose with throttle valve and electrolyte inlet

Proceed accordingly with the pH electrode:

1. Plug the pH electrode cable in the socket PH of the connector panel on the backside of SurPASS 3 (pos. 1 in Fig. 6).

2. Place the pH electrode in an appropriate hole of the beaker cover (pos. 1 in Fig. 15).

---

**WARNING**

Risk of injury

When moving SurPASS 3 from cold to warm surroundings, parts may become conductive due to water condensation. Strictly obey all grounding regulations!

Interrupting the protective grounding inside or outside of SurPASS 3 is inevitably dangerous. Never use extension cables without a protective ground connector.

**NOTICE**

The beaker cover has been designed for use with a 250 ml glass beaker (60 mm diameter) which is supplied with the SurPASS 3 instrument.
3.7 Mounting the Hoses of the Titration Unit (not applicable to SurPASS 3 Eco)

Two syringe pumps (syringe pump A and B) for automated dispense of titration liquid are integrated in the SurPASS 3 instrument. The corresponding 1 ml syringes are already mounted on the SurPASS 3 upon delivery. Follow the instructions given in chapter 6.8 if you need to remove and remount them for e.g. maintenance purposes or replacement.

Two sets of hoses are supplied with the SurPASS 3 accessories:
- 2 dispense lines (red and blue) with connector and ferrules on one end, and burette tip on the other end,
- 2 uptake lines (red and blue) with connector and ferrules on one end only

These hoses need to be mounted on the top left and right positions of the 3-way valves of the integrated titration unit.

1. Mount the nut of the dispense line in the top-left hole with thread of a 3-way valve.
2. Place the burette tip of a dispense line in the appropriate hole of the beaker cover (pos. 3 in Fig. 15)
3. Mount the nut of an uptake line in the top right hole with thread of a 3-way valve.
4. Fix the open end of the uptake line in the supply bottle with the titration liquid.

**WARNING**

**Risk of injury**
Handle the hoses for the titration unit only when they are clean and empty.

**CAUTION**

**Hose connections**
For a safe operation check the titration hoses and connections regularly.

**TIP:** To use the settings in the SurPASS 3 software, connect acid to syringe pump A and base to pump B.

5. Proceed accordingly with the second titration unit.

3.8 SurPASS 3 Accessory Kit for Liquid Handling

The SurPASS 3 accessory kit for liquid handling provides a conscientious and safe supply of the used liquids. This accessory arranges the two supply bottles for the titration liquids, a 400 ml waste beaker with cover for connecting the outlet hose and two storage containers for the pH and conductivity electrodes, respectively.

Mount the SurPASS 3 accessory kit for liquid handling as shown in Fig. 17. Place the device on the right side of the SurPASS 3 instrument close to the titration unit. Make sure the hoses of the titration unit are mounted as described in chapter 3.7.
3 Preparing the Instrument

3.9 SurPASS 3 Pressure Control Unit
(not applicable to SurPASS 3 Eco)

**WARNING**

*Risk of injury*
To avoid a flooding after the clean cycle, empty the waste beaker and the dike if there is any liquid in it.

**TIP:** Make sure that the nitrogen bubbles gently during the measurements to avoid the dissolution of the CO$_2$ from air.

---

1. Connect the end of hose (2) with the orange throttle valve (in direction of the arrow towards the beaker) to the electrolyte inlet to the T-connector. The other end of hose (2) still needs to be connected to the position "PURGE" of the pressure control unit (1). Mount the hose tightly into the connector by pushing it in until the end stop.

2. Upon delivery of the SurPASS 3 instrument, hose (3) is already connected between the position "OUT" of the pressure control unit (1) and the gas hose connector (position "p") on the instrument.

3. The CAN cable (4) connects the right one of the two sockets at the lower position of the pressure control unit (1) with the socket "CAN" of the connector panel on the backside of the SurPASS 3 instrument (pos. (5) in Fig. 6).

4. Hose (5) needs to be connected between the position "IN" on the pressure control unit (1) and the nitrogen supply in your laboratory. Refer to appendix C for details on the requirements for nitrogen supply and gas consumption.
3.10 SurPASS 3 Software

3.10.1 Installation of the SurPASS 3 Software

For the installation of the SurPASS 3 software start the installation file and follow the instructions of the setup program. Before installing the software, read the system requirements and make sure that your PC or laptop computer meets the hardware and software requirements (see chapter 2.3).

**TIP:** You must have administrator privileges for the installation of the SurPASS 3 software.

**NOTICE**

The operating pressure of the pressure control unit, i.e. the pressure of the nitrogen supply in your laboratory, shall be set to 6 bar. The operating pressure must not exceed 10 bar. The outgoing pressure is adjusted in the software.

The following software components will be automatically installed upon first installation of the SurPASS 3 software:

- Microsoft Windows Installer 4.5
- Microsoft .NET Framework 4.5.1

You do not have to enter a license key for operation of SurPASS 3 with the SurPASS 3 software.

3.10.2 Graphical User Interface

Open the SurPASS 3 software by double-clicking on the icon, which was created on the PC desktop during installation.

The SurPASS 3 software is a Microsoft Windows® based software offering the standard features of Windows programs.

Software functions specific for the operation of the SurPASS 3 instrument are (numbers in brackets refer to Fig. 19) described below:

**TIP:** Certain software features are disabled if you are operating a SurPASS 3 Eco.

- **Application Button (1):** Click on the application button <Sp> to open the settings view.
- **Settings View:** In the settings view you can select between "Preferences", "About" and "Software update":
  - **Preferences:** Use the drop-down menus to define the preferred unit settings for each parameter. The software needs to be restarted to activate any changes in the settings.
  - **About:** This field gives information about the currently installed software and firmware versions. It allows for starting a firmware update and creating a diagnostics package.
  - **Software Update:** Use this function to download software updates. If connected to the internet, the PC displays an alert ‘!’ in the quick access toolbar when a new software version is released.
- **Quick Access Toolbar (2):** Click this button to create a new experiment.
- **Experiment Menu (4):** The experiment menu is already expanded when starting the software. In the experiment menu, new experiments can be generated. The experiment menu consists of the "Experiment Name", which is typically best defined by the date of experiment and the name of the sample. In the field "User", the user can enter his or her name. The "Location" folder can be changed by clicking on the <...> button. The "Comments" field can be used for any entries concerning the experiment, e.g. information on sample preparation, availability of nitrogen purge or other information.

The experiment menu can also be folded and unfolded by clicking on the blue tab "Experiment". A new experiment can be opened by a click on the button <Set up new Experiment> in the quick access toolbar.

- **Current Device Configuration (21):** The current device configuration displays the information on all measured parameters in one single page.
3 Preparing the Instrument

• **Measurement Types**: You can select between six different measurement types:
  - **Dosage (8), not available with SurPASS 3 Eco**: Use the dosage function to either adjust the pH value of the electrolyte solution to a specific pH using one of the titration units, or to dispense a specific volume of liquid by the titration units.
  - **Rinse (9)**: Use the rinse function to fill the measuring cell and to rinse the sample with electrolyte solution before starting a zeta potential measurement.
  - **Zeta Potential (10)**: Use the zeta potential function to start a zeta potential measurement at the given electrolyte composition.
  - **Isoelectric Point (11), not available with SurPASS 3 Eco**: Use the isoelectric point measurement mode to determine the isoelectric point fully automatically. For each pH step max. 4 zeta cycles are selectable.
  - **pH Scan (12), not available with SurPASS 3 Eco**: The pH scan allows to perform a pH dependent zeta potential measurement until a user-defined end pH value is achieved. For each pH step max. 4 zeta cycles are selectable.
  - **Adsorption Kinetics (13), not available with SurPASS 3 Eco and only accessible if external pressure supply is activated**: The adsorption kinetics measurement type allows to monitor fast and slow adsorption processes. "Fast adsorption" stops after 3-4 cycles, depending on the gap height. "Slow adsorption" is used for adsorption processes where a longer measurement time is needed.

• **Start Button (20)**: Use the <start button> to start the selected measurement type.

• **Measurement View (5)**: Select the measurement view to monitor the performance of the pressure ramps during measurement or to control the gap adjustment by using the time during rinse cycles.

• **Result View (6)**: Select the result view to display the result of your zeta potential measurement.

• **Clean (22)**: A click on <Clean> opens the cleaning procedures with the following functions:
  - **Empty the system**: The task is used for pumping air through the measuring cell and thus removing any liquid after a measurement has finished.
  - **Clean the system**: This task is used for cleaning the electrolyte system with the cleaning device mounted.
  - **Fill the titration units (not available with SurPASS 3 Eco)**: This task is used for filling the hoses of the titration unit with acid and base.

• **External Pressure Supply (19)**: Click on the symbol to activate or deactivate the external pressure supply and thus the accessibility of the adsorption kinetics feature. The status of the external pressure supply is displayed as green or gray sign.

• **Maintenance (23)**: A click on <Maintenance> allows to select between:
  - **Conductivity Calibration**: The conductivity calibration shall be performed once a month. A message will be highlighted when the calibration is due.
  - **pH Calibration**: The pH calibration shall be performed once a week. A message will be highlighted when the calibration is due.
  - **Liquid Declaration (not available with SurPASS 3 Eco)**: Assign acid and base to syringe pumps A and B of the titration unit for a correct functioning of measurement types Isoelectric Point and pH Scan.

• **SurPASS 3 Viewer (3)**: Click on the <SurPASS 3 Viewer> symbol to open a new SurPASS 3 viewer instance or to bring the viewer to the foreground.

---

**WARNING**

**Risk of injury**

This process must be started with mounted measuring cell with Gap Height/Permeability Index adjusted. If you start the empty process with open gap or the cleaning device mounted, the liquid will splash out of the beaker cover.
3 Preparing the Instrument

Fig. 19: Graphical user interface of the SurPASS 3 software

1 Application button
2 Quick Access Toolbar for starting a new experiment
3 SurPASS 3 viewer
4 Experiment menu
5 Measurement view
6 Result view
7 Device status
8 Dosage
9 Rinse
10 Zeta Potential
11 Isoelectric Point
12 pH Scan
13 Adsorption Kinetics
14 Selection between voltage and current measurement
15 Tag to identify measurement file
16 Measurement parameters
17 Advanced measurement options
18 Number of measuring cycles
19 Status of the external pressure supply
20 Start button
21 Current device configuration
22 Clean tasks (Empty, Clean and Fill)
23 Maintenance
24 pH calibration reminder

3.10.3 Export of Data

All measured data are automatically saved in *.xlsx- and *.csv-files. By default result files are saved in the folder C:UsersusernameDocumentsSurPASS3AuthorExperiment (refer also to chapter 5.6).

TIP: The content of the respective *.xlsx- and *.csv-file is identical, except for the unit settings (customized unit settings in *.xlsx-file, SI units in *.csv-file).

The *.csv-file is needed by the SurPASS 3 viewer and must not be deleted.

SurPASS 3 software serves primarily for the purpose of instrument operation. Measurement files can be re-opened in the SurPASS 3 software, and are then displayed in the SurPASS 3 viewer. A spreadsheet software needs to be used for evaluation of measured data.
4 Putting SurPASS 3 into Operation

4.1 pH Electrode Calibration

The calibration of the pH electrode requires three buffer standard solutions of known pH value. The buffer standard solutions for calibrating the pH electrode should equally cover the complete measuring range (e.g. pH 3, pH 7, pH 10). To ensure an accurate calibration and pH measurement it is important to use fresh buffer solutions for the calibration.

1. Click on <Maintenance> and open the task "pH calibration".

TIP: If the message for the need for pH calibration is displayed, a click on the message guides you to the task "pH calibration".

![Fig. 20: pH calibration reminder](image)

2. Click on <Begin calibration>.

3. Clean the pH electrode with ultrapure water and insert it into the first buffer solution.

4. Enter the pH value of this first buffer solution in the first number field. The measured voltage and pH value will be displayed. Click on the small triangle next to "Start" to start the pH calibration.

5. The progress bar indicates the progress of the measurement. Wait until it has finished, then clean the pH electrode with water and place it into the next buffer solution. Enter its pH value into the second number field and click on the <Start> button to continue the pH calibration.

6. Proceed accordingly with the third buffer solution.

7. If the pH calibration parameters are accepted by the software, click on <Accept> to save them. A green checkmark will indicate a successful calibration.

8. If any of the values is out of range, click on <New calibration> to repeat the pH calibration, or click on <Close> to repeat the pH calibration at a later time.

Possible reasons for an unsuccessful pH calibration are:

- an invalid pH of one of the buffer standard solutions (consider a temperature effect on pH),
- a drift of the pH electrode or
- a damage of the pH electrode connector or cable.

NOTICE

- It is recommended to calibrate the pH electrode once a week. A message will be highlighted when the calibration is due.
- If not in use store the pH electrode in 3 mol/l KCl solution and place it in the storage container of the accessory kit for liquid handling (pos. 5 in Fig. 17).

NOTICE

At 25 °C the following values for the pH calibration parameters Slope and Offset are acceptable: Slope: -59.16 mV/pH ± 4 % (-56.8 mV/pH to -61.5 mV/pH) Offset: ± 30 mV
4.2 Conductivity Electrode Calibration

The calibration of the conductivity electrode is a single point calibration and requires a 0.1 mol/l KCl solution or a conductivity standard solution.

The conductivity electrode delivered with the SurPASS 3 contains a temperature sensor. The temperature measurement by this sensor is used for both conductivity calibration and zeta potential evaluation.

1. Click on <Maintenance> and open the task "Conductivity calibration".

TIP: If the message for the need for conductivity calibration is displayed, a click on the message guides you to the task "conductivity calibration".

2. Click on <Begin calibration>.

3. Choose a 0.1 mol/l solution of KCl or a conductivity standard for conductivity calibration.

4. Prepare 200 ml of the 0.1 mol/l KCl solution or conductivity standard solution in the 250 ml glass beaker.

4.2.1 Conductivity Calibration using 0.1 mol/l KCl

1. Select "0.1 mol/l KCl" as calibration liquid.

2. Clean the conductivity electrode with ultrapure water and insert it into the 250 ml glass beaker filled with 0.1 mol/l KCl solution until the hole in the shaft is immersed completely. The measured conductivity and temperature will be displayed.

3. Click on the small triangle next to "Start" to start the conductivity calibration.

4. The progress bar indicates the progress of the measurement. Wait until it has finished.

5. If the cell constant is accepted by the software, click on <Accept> to save it. A green checkmark will indicate a successful calibration.

6. If the cell constant is unacceptable, click on the small triangle to start the calibration again, or click on <Close> to repeat the conductivity calibration at a later time.

TIP: The conductivity electrode supplied with the SurPASS 3 instrument has a cell constant of $47.5 \text{ m}^{-1} \pm 5.25\%$ ($45 \text{ m}^{-1}$ ... $50 \text{ m}^{-1}$). A persistent deviation by $> 5.25\%$ indicates the needs for exchanging the conductivity electrode.

TIP: Watch the correct unit for conductivity when using a conductivity standard.

4.2.2 Conductivity Calibration using a Conductivity Standard

1. When using a conductivity standard, select "conductivity standard" and enter the name of the conductivity standard and its conductivity at the given temperature as stated on the bottle.

2. Proceed with steps 2-6 in chapter 4.2.1.

TIP: The conductivity electrode supplied with the SurPASS 3 instrument has a cell constant of $47.5 \text{ m}^{-1} \pm 5.25\%$ ($45 \text{ m}^{-1}$ ... $50 \text{ m}^{-1}$). A persistent deviation by $> 5.25\%$ indicates the needs for exchanging the conductivity electrode.

TIP: Watch the correct unit for conductivity when using a conductivity standard.
4.3 Filling of the Titration Unit (not applicable to SurPASS 3 Eco)

The SurPASS 3 instrument with titration unit contains two syringes with a volume of 1.0 ml for dispensing acid and base to change the pH of the electrolyte solution.

The hoses of the titration unit have to be filled before starting a pH titration. If different titration liquids are in use, the hoses have to be rinsed after exchanging the titration liquid. Make sure the titration hoses are mounted properly before proceeding with filling the titration units.

1. In order to assign the liquids in use to the respective syringe pump, select <Maintenance> and "Liquid Declaration".
2. Select "Acid", "Base" or "Other" for each syringe pump and save these settings.

**TIP:** "Acid" or "Base" needs to be selected in order to enable isoelectric point or pH scans, respectively.

3. In order to fill the titration unit, select <Clean> and "Fill the titration units".
4. Click on <Start> to start the filling of the titration units. The system uses a volume of 4 ml for each syringe pump to ensure proper filling of the hoses.

**TIP:** The same procedure can be used for cleaning and emptying the titration unit. Use ultrapure water for cleaning and put the dispense line in a waste beaker. Remove the uptake line from the liquid reservoir to empty the titration unit by performing fill cycles with air.

4.4 SurPASS 3 Verification Measurement with Cotton Fabrics

Follow these instructions in order to perform a verification measurement using a standard cotton fabric (ISO 105-F09) on your SurPASS 3 instrument. Meeting the specified range of the zeta potential of this cotton fabric confirms that the SurPASS 3 is in a reliable working condition.

4.4.1 Chemicals and Accessories

In order to perform the verification measurement, you need:

1. the accessory for SurPASS 3 verification measurement (cat.no. 181830) incl. a set of standard fabrics for verification measurement (cat.no. 182927)
4.4.2 Preparation of the 0.01 mol/l KCl Standard Solution for the Verification Measurement with Ag/AgCl Electrodes

1. Rinse the 250 ml volumetric flask that is contained in the accessory for SurPASS 3 verification measurement with ultrapure water.
2. Add the amount of KCl required to prepare the 0.01 mol/l standard solution, i.e. 0.186 g for 250 ml.
3. Transfer the KCl salt into the volumetric flask. Make sure that the complete amount of KCl is transferred.
4. Fill the volumetric flask with ultrapure water exactly up to the ring marker.
5. Close the volumetric flask and tilt it several times to guarantee a homogeneous mixing of the salt with the ultrapure water.

### Table 1: Preparation of the 0.01 mol/l KCl Standard Solution

<table>
<thead>
<tr>
<th>Volume of the volumetric flask [ml]</th>
<th>Amount of KCl [g]</th>
<th>Amount of KCl [mg]</th>
</tr>
</thead>
<tbody>
<tr>
<td>250a</td>
<td>0.186</td>
<td>186</td>
</tr>
<tr>
<td>500</td>
<td>0.373</td>
<td>373</td>
</tr>
<tr>
<td>1000</td>
<td>0.746</td>
<td>746</td>
</tr>
</tbody>
</table>

*a Contained in the accessory for SurPASS 3 verification measurement*

4.4.3 Preparation of the 0.001 mol/l KCl Standard Solution for the Verification Measurement with Gold Electrodes

1. Rinse the 250 ml volumetric flask that is contained in the accessory for SurPASS 3 verification measurement with ultrapure water.
2. Add the amount of KCl required to prepare the 0.001 mol/l standard solution.
3. Transfer the KCI salt into the volumetric flask. Make sure that the complete amount of KCI is transferred.
4. Fill the volumetric flask with ultrapure water exactly up to the ring marker.
5. Close the volumetric flask and tilt it several times to guarantee a homogenous mixing of the salt with the ultrapure water.

### Table 2: Preparation of the 0.001 mol/l KCl Standard Solution

<table>
<thead>
<tr>
<th>Volume of the volumetric flask [ml]</th>
<th>Amount of KCl [g]</th>
<th>Amount of KCl [mg]</th>
</tr>
</thead>
<tbody>
<tr>
<td>250a</td>
<td>0.0186</td>
<td>18.6</td>
</tr>
<tr>
<td>500</td>
<td>0.0373</td>
<td>37.3</td>
</tr>
<tr>
<td>1000</td>
<td>0.0746</td>
<td>74.6</td>
</tr>
</tbody>
</table>

*a Contained in the accessory for SurPASS 3 verification measurement*

4.4.4 Mounting the Standard Cotton Fabric

1. Take one standard cotton fabric and fold it four times. You get a square sample of 1 cm x 1 cm with 16 cotton fabric layers.

3. Make sure that no fibers are blocking the thread when closing the sample holder.

4. Use the two mounting tools to tighten the sample holder completely, so that no gap is visible between the two halves.

5. Mount the sample holder into the SurPASS 3 verification device. Fix the screws by using the Torx T20 screwdriver with torque control.

6. Mount the SurPASS 3 verification device on the instrument as described in chapter 5.1.

7. Transfer 200 ml of the 0.01 mol/l KCl (or 0.001 mol/l KCl) volumetric electrolyte solution into the 250 ml glass beaker.

8. The conductivity of the 0.01 mol/l KCl (or 0.001 mol/l KCl) standard solution has to match the tabulated data. A deviation of < 5 % is acceptable.
4.4.5 Verification Measurement with Standard Cotton Fabric

1. Start a new experiment in the SurPASS 3 software as described in chapter 5.2.

2. Start 5 rinse cycles with the default parameters:
   - Fill volume: 100 ml
   - Start pressure: 600 mbar
   - Stop pressure: 200 mbar
   - Rinse cycles: 5

3. Verify that the permeability index is in the range of 100 ... 130.

4. Start 5 Zeta Potential measurements with the default parameters as described in chapter 5.3.1:
   - Fill volume: 100 ml
   - Start pressure: 600 mbar
   - Stop pressure: 200 mbar
   - Zeta cycles: 5
   - Activate “Advanced measurement options”
   - Upper pressure limit: 450 mbar

4.4.6 Verification Results for Ag/AgCl Electrodes

Use the results of measurements no. 3, no. 4 and no. 5 to evaluate the zeta potential of the standard cotton fabric. The expected value for the zeta potential is -19 mV. A deviation of ±10 % is acceptable. The corresponding pressure ramp data (streaming potential vs. pressure difference) shall be linear and overlapping.

TIP: Refer to the reference guide D85IB001EN for more information, tips and tricks in case the verification measurement did not yield the expected result.

4.4.7 Verification Results for Gold Electrodes

Use the results of measurements no. 3, no. 4 and no. 5 to evaluate the zeta potential of the standard cotton fabric. The expected value for the zeta potential is -15 mV ± 2 mV.

---

### Table 4-1: Conductivity data

<table>
<thead>
<tr>
<th>temperature in °C</th>
<th>0.01 mol/l KCl solution conductivity in mS/m</th>
<th>conductivity in µS/cm</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>127.6</td>
<td>1276</td>
</tr>
<tr>
<td>21</td>
<td>130.4</td>
<td>1304</td>
</tr>
<tr>
<td>22</td>
<td>133.1</td>
<td>1331</td>
</tr>
<tr>
<td>23</td>
<td>135.8</td>
<td>1358</td>
</tr>
<tr>
<td>24</td>
<td>138.6</td>
<td>1386</td>
</tr>
<tr>
<td>25</td>
<td>141.3</td>
<td>1413</td>
</tr>
<tr>
<td>26</td>
<td>144.1</td>
<td>1441</td>
</tr>
<tr>
<td>27</td>
<td>146.8</td>
<td>1468</td>
</tr>
<tr>
<td>28</td>
<td>149.6</td>
<td>1496</td>
</tr>
<tr>
<td>29</td>
<td>152.4</td>
<td>1524</td>
</tr>
<tr>
<td>30</td>
<td>155.0</td>
<td>1550</td>
</tr>
</tbody>
</table>

Source: Handbook of Chemistry and Physics

9. The pH of the 0.01 mol/l KCl standard solution has to fit within the limits of pH 5.5 - pH 6.5.
The corresponding pressure ramp data (streaming potential vs. pressure difference) should be linear and overlapping.

Fig. 40: Pressure ramps with gold electrodes
5 Performing a Measurement

NOTICE
The SurPASS 3 was tested and cleaned before shipment. It is not necessary to rinse or clean the instrument before starting the first measurement after installation.

NOTICE
Make sure to use ultrapure water (ASTM I) and high purity chemicals as described in chapter 2.4 before starting the first measurement.

In order to perform a successful zeta potential analysis with SurPASS 3, the following procedures have to be performed in the given order:

1. Mount the sample in a suitable measuring cell (refer to the reference guide D85IB001EN).
2. Mount the measuring cell on SurPASS 3 (see chapter 5.1).
3. Define a new experiment in the software and rinse the system with electrolyte solution (see chapter 5.2).
4. Adjust the gap height if one of the Adjustable Gap Cells or the Clamping Cell is used (see chapter 5.2.1).
   Adjust the permeability index if the Cylindrical Cell is used (see chapter 5.2.2).
5. Define the parameters for either a zeta potential analysis, isoelectric point determination or pH scan and start the measurement (see chapter 5.3).
6. After finishing the measurement, empty the system (see chapter 5.5.1).
7. Remove the measuring cell with the sample and mount the cleaning device (see chapter 5.5.2).
8. Clean the system by rinsing the electrolyte circuit with ultrapure water or an appropriate cleaning solution (see chapter 5.5.3).

5.1 Mounting the Measuring Cells on SurPASS 3

1. Observe the correct positioning of the measuring cell with the arrow always pointing upwards.

NOTE
The SurPASS 3 was tested and cleaned before shipment. It is not necessary to rinse or clean the instrument before starting the first measurement after installation.

NOTICE
Make sure to use ultrapure water (ASTM I) and high purity chemicals as described in chapter 2.4 before starting the first measurement.

In order to perform a successful zeta potential analysis with SurPASS 3, the following procedures have to be performed in the given order:

1. Mount the sample in a suitable measuring cell (refer to the reference guide D85IB001EN).
2. Mount the measuring cell on SurPASS 3 (see chapter 5.1).
3. Define a new experiment in the software and rinse the system with electrolyte solution (see chapter 5.2).
4. Adjust the gap height if one of the Adjustable Gap Cells or the Clamping Cell is used (see chapter 5.2.1).
   Adjust the permeability index if the Cylindrical Cell is used (see chapter 5.2.2).
5. Define the parameters for either a zeta potential analysis, isoelectric point determination or pH scan and start the measurement (see chapter 5.3).
6. After finishing the measurement, empty the system (see chapter 5.5.1).
7. Remove the measuring cell with the sample and mount the cleaning device (see chapter 5.5.2).
8. Clean the system by rinsing the electrolyte circuit with ultrapure water or an appropriate cleaning solution (see chapter 5.5.3).

5.1 Mounting the Measuring Cells on SurPASS 3

1. Observe the correct positioning of the measuring cell with the arrow always pointing upwards.

CAUTION
Risk of injury
Watch your fingers when fixing the measuring cell.

4. The SurPASS 3 software will recognize that a measuring cell is connected and the name of the measuring cell will be displayed in the current device configuration.
5.2 Rinsing the System

**NOTICE**
- Make sure the complete electrolyte circuit and the measuring cell were rinsed with ultra-pure water (ASTM I) before starting a measurement.
- Make sure the pH and conductivity electrodes are calibrated.

1. Check the proper connection of the external electrolyte hoses (see chapter 3.5).

**NOTICE**
The water used for rinsing and for preparing the electrolyte solution for zeta potential measurement must conform with the American Society for Testing and Materials standard ASTM I.

**NOTICE**
Do not clean the glass bottles used with SurPASS 3 in the dishwasher. Residues of detergents are difficult to remove from the SurPASS 3 electrolyte circuit and can falsify the zeta potential results.

2. Put the magnetic stir bar and fill 200 ml of electrolyte solution into the glass beaker.

3. If required start purging the electrolyte solution with nitrogen. For details on the requirements for nitrogen supply refer to appendix C. Make sure that the external pressure is activated in the software as indicated by a green external pressure supply sign (refer to chapter 3.10.2).

4. Create a new experiment in the SurPASS 3 software by filling out the fields "Experiment Name" and "User". The file "location" can be altered by clicking on <...> and selecting a different folder. Any comments on the experiment can be entered and the new experiment is started by clicking on <Set up new Experiment>.

5. Select the task "Rinse". Use the default values of 100 ml as fill volume, 600 mbar as start pressure, 200 mbar as stop pressure and 3 cycles.

**TIP:** Typically, the name of the sample is an appropriate name for the experiment.

**TIP:** The new experiment menu is already open when starting the software. If you need to open the menu manually, click on the <Set up new Experiment> button in the quick access toolbar.

6. Click on <Start>.

7. Double-click into the graph for full display of the data. Follow the performance of the pressure ramps ramp (pressure vs. time) in the measurement view.

8. If necessary adjust the gap height or the permeability index during the rinse cycles.

**TIP:** Make sure that nitrogen is available and that the external pressure is activated in the software (refer to chapter 3.10.2) if the start pressure needs to be set higher than 1500 mbar.

**TIP:** If the streaming resistance (refer to chapter 5.2.1) is extremely high, residues of air are remaining in the measuring cell. To get rid of the air, open the gap by using the adjustment knob (for Adjustable Gap Cell and Clamping Cell) during the rinse cycle and then close it again.

**TIP:** The defined values for start and stop pressure are also used for the rinsing cycles during the "Isoelectric Point" and "pH Scan" measurement.

**NOTICE**
- Make sure the complete electrolyte circuit and the measuring cell were rinsed with ultra-pure water (ASTM I) before starting a measurement.
- Make sure the pH and conductivity electrodes are calibrated.

**NOTICE**
The water used for rinsing and for preparing the electrolyte solution for zeta potential measurement must conform with the American Society for Testing and Materials standard ASTM I.

**NOTICE**
Do not clean the glass bottles used with SurPASS 3 in the dishwasher. Residues of detergents are difficult to remove from the SurPASS 3 electrolyte circuit and can falsify the zeta potential results.
5 Performing a Measurement

5.2.1 Adjusting the Gap between Planar Sample Surfaces

1. Monitor the pressure drop over time which is displayed continuously in the current device configuration. Adjust the gap height by rotating the adjustment knob.

**Fig. 45: Rinse cycles**

**TIP:** With the standard measurement parameters, the end pressure of 200 mbar should be reached in approx. 65 to 75 seconds so that your alignment is in the range of 80 to 120 µm.

2. Monitor the gap height which is displayed in the current device configuration after each rinse cycle has finished.

**Fig. 46: Gap height**

3. According to this gap height,
   a. use the adjustment knob to close/open the gap carefully to achieve the target gap of 100 µm (1 unit of the scale on the adjustment knob equals 25 µm). If necessary start additional rinse cycles.
   b. close the adjustment knob by min. 300 µm, if no gap is displayed
   c. open the adjustment knob if the pressure displayed in the current device configuration does hardly decrease (streaming resistance is extremely high).

4. Repeat steps 2-3 until the gap height is adjusted to 100 µm.

5.2.2 Adjusting the Permeability Index of Sample Plugs in the Cylindrical Cell

1. Monitor the permeability index which is displayed in the current device configuration after the rinse cycle has finished.

2. Adjust the permeability index approximately to 100 by turning the micrometer screw.

5.3 Starting a Zeta Potential Measurement

The routines "Rinse" (chapter 5.2), "Zeta Potential" (chapter 5.3.1), "Isoelectric Point" (chapter 5.3.2) and "pH Scan" (chapter 5.3.3) record pressure ramps. Fill volume, start pressure and stop pressure can be defined for each of these routines. The default settings (Fill volume 100 ml, Start pressure...) can be used for most analyses.

5.3.1 Zeta Potential

1. Select the measurement type "Zeta Potential" to perform a single or a series of zeta potential measurements at defined electrolyte composition.

**TIP:** If you need to change the pH value of your electrolyte solution, first select the task "Dosage" to either add acid or base until a certain pH value is reached or to add a certain volume of acid or base. Make sure to insert the corresponding burette tip into the electrolyte beaker before clicking on <Start>. Perform at least two rinse cycles (see chapter 5.2) before starting a zeta potential measurement after changing the pH value.

2. Choose one (1) cycle if you wish to perform a single zeta potential measurement, or type in a higher number of measurements (up to 99) for a series of zeta potential measurements at same electrolyte composition.

3. Click on the <Start> button.
5 Performing a Measurement

1. Select the measurement type "Isoelectric Point" for the most efficient way to determine the isoelectric point of your sample.

2. Type in a number of rinse cycles which the instrument shall execute after the pH value was adjusted and before performing another zeta potential measurement at changed electrolyte composition. The start and stop pressure, defined in "Rinse", are used for these rinse cycles (refer to chapter 5.2).

**TIP:** Three rinse cycles are sufficient for most samples. If you observe from the measurement view that the pressure ramps are not overlapping after three cycles, stop the measurement and use a higher number of cycles to ensure equilibrium conditions for correct zeta potential determination.

3. Type in a number of zeta cycles which the instrument shall execute at the adjusted pH value. A max. of 4 repetitions are possible.

4. For the measurement the values for start and stop pressure defined in "Zeta Potential" are used.

5. The pH increment is set by default to 0.3 pH units and is adjustable up to 2 pH units.

6. Wipe off the burette tip of both titration units and place them into the electrolyte beaker. Make sure the titration hoses are filled with the respective titration liquids (refer to chapter 4.3).

7. Click on the <Start> button.

8. Monitor the performance of the pressure ramps in the measurement view during measurement by a double-click in the graph.

9. Find the zeta potential vs. pH dependence and the isoelectric point in the result view after the measurement has finished.

---

**WARNING**

**Hazardous Liquids**
The volume of electrolyte solution in the beaker must not exceed the 200 ml mark of the beaker when starting an Isoelectric Point or pH Scan to avoid an overflow of liquid during the course of the analysis.

---

4. Monitor the performance of the pressure ramps in the measurement view during measurement by a double-click in the graph.

5. The performance of the pressure ramps is very important, since the software is doing a linear fit through the recorded streaming current / streaming potential values. If necessary use the Advanced measurement options to define the upper pressure limit.

6. Find the zeta potential result in the current device configuration and in the result view after the measurement has finished.

**5.3.2 Isoelectric Point (not applicable to SurPASS 3 Eco)**

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**Fig. 47: Zeta potential measurement**

**Fig. 48: Pressure ramps (measurement view)**

**Fig. 49: Advanced measurement options**

**Check the box "Advanced measurement options" and type in the upper pressure where the recorded pressure ramp still appears linear, e.g. 450 mbar.**

**Fig. 50: Isoelectric point determination**

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5.3.3 pH Scan (not applicable to SurPASS 3 Eco)

**WARNING**

**Hazardous Liquids**
The volume of electrolyte solution in the beaker must not exceed the 200 ml mark of the beaker when starting an Isoelectric Point or pH Scan to avoid an overflow of liquid during the course of the analysis.

1. Select the measurement type "pH scan" for zeta potential determination in a defined pH range.
2. Type in a number of rinse cycles which the instrument shall execute after the pH value was adjusted and before performing another zeta potential measurement at changed electrolyte composition. The start and stop pressure, defined in "Rinse", are used for these rinse cycles (refer to chapter 5.2).

**TIP:** *Three rinse cycles are sufficient for most samples. If you observe from the measurement view that the pressure ramps are not overlapping after three cycles, you may want to stop the measurement and use a higher number of cycles to ensure equilibrium conditions for correct zeta potential determination.*

3. Type in a number of zeta cycles which the instrument shall execute at the adjusted pH value. A max. of 4 repetitions are possible.
4. For the measurement the values for start and stop pressure defined in "Zeta Potential" are used.
5. Type in the end pH value where you want the measurement to stop. The instrument will perform zeta potential measurements starting from the pH value of the electrolyte solution until the end pH value was passed. The pH increment is set by default to 0.3 pH units and is adjustable up to 2 pH units.
6. Wipe off the burette tip of the titration unit which is filled with acid or base, depending on the just defined end pH value, and place it into the electrolyte beaker. Make sure the titration hoses are filled with the respective titration liquid (refer to chapter 4.3).

7. Click on the <Start> button.

**Fig. 51: pH scan**

8. Monitor the performance of the pressure ramps in the *measurement view* during measurement by a double-click in the graph.
9. Find the zeta potential vs. pH dependence in the *result view* during and after the measurement has finished.

5.4 Adsorption Kinetics Measurement (not applicable to SurPASS 3 Eco)

Please refer to the reference guide D85IB001EN for more information about adsorption kinetics measurement.

5.5 End of Measurement

A measurement has finished when the <Stop> button turns into a <Start> button again.

5.5.1 Empty the System

1. Click on <Clean> and select the task "Empty the system" in the *cleaning procedures*.
2. Leave both the outlet and the inlet hoses in the beaker with the electrolyte solution.
3. Click on <Start> to pump air through the measuring cell and thus remove all liquid from the electrolyte circuit.

**NOTICE**
Avoid starting an empty process while the cleaning device or a cell with too large gap height is mounted as the measurement solution will splash out of the beaker.

### 5.5.2 Removing the Measuring Cell

**WARNING**

*Risk of injury*
Do not remove the measuring cell while the instrument is in operation.

**CAUTION**

*Hazardous liquids*
Always empty the electrolyte circuit of SurPASS 3 before removing the measuring cell.

1. Turn the locking lever on the front panel to the left.

### 5.5.3 Clean the System

1. Lift the cover or the 250 ml electrolyte beaker and connect the outlet hose (on the right side) to the beaker cover of the waste beaker.

2. Fill 200 ml of ultrapure water or cleaning solution into the 250 ml beaker.
5 Performing a Measurement

5.5.4 Switching off the Instrument

**NOTICE**

Before switching off the SurPASS 3 instrument, make sure the electrolyte system is clean and empty (refer to chapter 5.5.3).

1. Switch off the nitrogen supply in your laboratory.
2. Switch off the instrument by using the power switch on the rear side of the SurPASS 3 instrument.
3. Store the pH electrode in 3 mol/l KCl solution and place it in the storage position of the liquid handling kit (Pos. 5 in Fig. 17).
4. Place the conductivity electrode in dry condition into its storage position of the liquid handling kit (Pos. 4 in Fig. 17).

**TIP:** If you first switch off the nitrogen supply but keep the external gas supply activated in the software and the instrument switched on, a strange noise will indicate that the system is depressurized. Switch off the instrument to stop the noise.

5.6 Data Analysis

5.6.1 Export of Measured Data

After a measurement has finished, all data is saved in the folder which was defined as file location upon creating the experiment. By default, this is:

C:\Users\username\Documents\SurPASS3\Author\Experiment name (for Windows 7)

C:\My computer\Documents\SurPASS3\Author\Experiment name (for Windows 8)

In this folder, you will find:

- for an individual zeta potential measurement:
  - *.xlsx- and *.csv-files with the results for each
zeta potential measurement
- a subfolder containing *.xlsx- and *.csv-files of the raw data for each measurement
- the*.surf-file for display of the measured data in the SurPASS 3 viewer (see reference guide D85IB001EN).

• for an isoelectric point determination:
  - *.xlsx- and *.csv-files of the complete IEP scan
  - a subfolder containing *.xlsx- and *.csv-files of the raw data for each zeta potential measurement
  - the*.surf-file for display of the measured data in the SurPASS 3 viewer (see reference guide D85IB001EN).

TIP: The content of the respective *.xlsx- and *.csv-file is identical, except for the unit settings (customized unit settings in *.xlsx-file, SI units in *.csv-file). The *.csv-file is needed by the SurPASS 3 viewer and must not be deleted.

• for a pH scan:
  - *.xlsx- and *.csv-files of the complete pH scan
  - a subfolder containing *.xlsx- and *.csv-files of the raw data for each zeta potential measurement
  - the*.surf-file for display of the measured data in the SurPASS 3 viewer (see reference guide D85IB001EN).
6 Upkeep and Cleaning

This chapter contains information on how to handle the instrument to ensure its smooth long-term operation.

- After a standby > 2 weeks, or after a series of different application measurements, it is recommended to perform the instrument verification as described in section 4.4 SurPASS 3 Verification Measurement with Cotton Fabrics.
- Please take note of section 4.1 and section 4.2 concerning the pH and conductivity electrode calibration:
  - The pH electrode should be calibrated once a week.
  - The conductivity electrode should be calibrated once a month.
- To assure a constant and high accuracy of your measurement, operate and store the instrument under the specified conditions and perform the recommended tasks regularly.
- Ensure that all environmental requirements described in section 1.1 General Safety Instructions (sub-section "Installation and use") as well as in section 2.3 System Requirements and section 2.4 Water Quality and Measurement Conditions are always fulfilled.
- Always handle the instrument with care. Even minor damage may cause serious measurement errors.
- Employ a regular optical check if there is no contamination on the outside of the instrument and perform an effective cleaning routine.

**WARNING**

*Risk of injury*

Repair and service activities exceeding the procedures described in this instruction manual may only be carried out by authorized personnel. In case of a malfunction of the SurPASS 3 instrument contact your local Anton Paar representative.

6.1 Software Administration

- Keep the delivered USB stick with the software delivered with the instrument in a safe place.
- Perform an update if the software indicates a new firmware or software version available in the cloud.
- Make sure that the used computer meets the needed specifications as described in appendix A Technical Data and in section 2.3 System Requirements.

6.2 Upkeep and Cleaning after Standard Operation

- Wipe the instruments cover to keep it free of dust and to avoid chemical damage on the instrument cover surface.
- Perform a clean cycle with ultra-pure water after the measurement operation and before switching off the instrument.
- It is not allowed to operate or clean the instrument with flammable liquids.
- Rinse the pH electrode with ultra-pure water and then put it in the appropriate storage beaker filled with KCL 3 mol/L if the instrument is not in use.
- Rinse the conductivity electrode with ultra-pure water and then put it in the storage beaker of the instrument in dry condition if the instrument is not in use.
- Turn off the nitrogen purge in the software and also the in-house nitrogen supply if not in use.
- Empty the electrolyte and the waste beakers if the instrument is not in use.
- Put the dispense lines for acid and base into the storage position if they are not in use.

6.3 Upkeep and Cleaning after Adsorption Operation

Clean the instrument using ultra-pure water or an appropriate cleaning solution.

To keep the measurement performance an extensive cleaning after the adsorption tests is recommended. Refer to section 6.7 Manual Cleaning of the Electrolyte Circuit.

**Note:** It is not allowed to clean the instrument with flammable liquids.
6.4 Cleaning and Drying the Measuring Cells

- The gaskets and O-rings of all measuring cells need to be checked after a measurement to guarantee trouble-free operation.
- If the cells are not in use, store them disassembled in dry condition.
- Make sure that they were cleaned with ultra-pure water and that all residues of samples are removed.

6.5 Wetted Parts

During the rinse and measurement processes the electrolyte gets in contact with the following materials.

- PharMed BPT tube (fluorinated polymer)
- Silicone tube
- PVDF poly(vinylidene fluoride)
- Borosilicate glass
- SiC Silicon carbide
- FPM/FKM (fluorinated rubber)
- EPDM ethylene propylene diene monomer
- POM-C (Polyoxymethylene Copolymer)
- SurPASS 3 adapter for cleaning (cleaning device)
  - PP polypropylene
- Cylindrical Cell
  - PA poly(amide)
  - PCTFE poly chloro trifluoro ethylene
  - PEEK polyether ether ketone
- Adjustable Gap Cell D14/D15
  - PPS polyphenylene sulfide
- Adjustable Gap Cell 20 x 10 mm
  - PEEK GF30 polyether ether ketone
- Titration Unit
  - ETFE ethylene tetrafluoro ethylene
  - PTFE poly(tetrafluoro ethylene)

6.6 Exchange of the Electrodes

With SurPASS 3 you may use Ag/AgCl or gold electrodes.

Please refer to the reference guide D85IB001EN for more information about exchanging of the electrodes.

6.7 Manual Cleaning of the Electrolyte Circuit

This chapter contains information on how to clean the electrolyte circuit manually in case of a severe...
6.8 Removing and Mounting of the Syringes of the Titration Unit (not applicable to SurPASS 3 Eco)

Follow the instructions given in the reference guide D85IB001EN in case you need to remove and remount the syringes of the titration unit.

6.9 Venting the Gas Supply Line (not applicable to SurPASS 3 Eco)

Proceed as follows to disconnect the SurPASS 3 instrument from your in-house gas supply:

1. Make sure the external gas supply is activated in the software.
2. Switch off the nitrogen supply in your laboratory.
3. Watch the display on the manometer of your in-house gas supply and wait for the pressure to drop. This might take 1-2 minutes.
4. Switch off the instrument by using the power switch on the rear side of the SurPASS 3 instrument.
5. Detach the pressure hose (hose (5) in Fig. 18) by pressing down and holding its releasing ring. Remove the hose.

6.10 Storing and Transporting the Instrument

- Make sure that it was rinsed with ultra-pure water and that it is free of salt and aggressive chemical liquids.
- All liquids and samples residues must be removed from the measuring cells if not in use.
- Empty and rinse the acid and base titration lines to keep up the titration syringes if the instrument is not in use.
- Turn off the in-house nitrogen supply and vent the gas supply line. Refer to section 6.9 Venting the Gas Supply Line (not applicable to SurPASS 3 Eco) for details.
- Empty the measuring cell before you move or lift the instrument.
- Store the instrument in a dry and empty condition.
- Store the pH electrode in the appropriate storage beaker filled with 3 mol/L KCl.
- Store the conductivity electrode in the storage beaker of the instrument in dry condition.
- The SurPASS 3 must be transported in the original package or in the corresponding transport box.
- During transport, the instrument must not be exposed to intense shocks or high forces.

6.11 Packing the Instrument for Returns

This should be performed by a qualified service technician. Contact your local Anton Paar representative for details.

**TIP:** Find the contact data of your local Anton Paar representative on the Anton Paar website (http://www.anton-paar.com) under "Contact".
7 Maintenance and Repair

7.1 Maintenance Performed by an Authorized Anton Paar Service Engineer

The SurPASS 3 requires a periodical maintenance which shall be performed by an authorized Anton Paar Service Engineer\(^1\).

A missing maintenance may mean that under certain conditions your warranty is no longer valid\(^2\).

**Maintenance Interval:**
- Once a year,
- Or if the standard maintenance does not lead to a successful verification,
- Whichever is sooner.

**Parts to be exchanged at every service interval (wear and tear parts):**
- Set of O-rings for the plug-in module (including hydraulic valve block, pressure vessel, front panel and hydraulic valve motor)
- O-rings of the electrodes
- Titration hoses and titration syringes if corroded
- Electrolyte hoses
- Nitrogen purge tube and throttle
- Gaskets and O-rings of the measuring cells

**Parts to be controlled at every service interval and to be exchanged if required (wear and tear parts):**
- Ceramic valve disks
- Membrane pump
- Digital pressure regulator
- Conductivity electrode
- pH electrode

7.2 Repair Performed by an Authorized Anton Paar Representative

In case your instrument needs repair, contact your local Anton Paar representative, who will take care of the necessary steps.

If your instrument needs to be returned, request an RMA (Return Material Authorization Number). It must not be sent without the RMA and the filled "Safety Declaration for Instrument Repairs". Please make sure it is cleaned before return.

**TIP:** Find the contact data of your local Anton Paar representative on the Anton Paar website (http://www.anton-paar.com) under "Contact".

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\(^1\) Please contact your Anton Paar representative to get an offer.

\(^2\) For detailed information please see the general terms of delivery (GTD) on the Anton Paar website (http://www.anton-paar.com).
## Appendix A: Technical Data

### Measuring range

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Range and Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>Streaming potential</td>
<td>± 2000 mV ± (0.2 % + 4 µV)</td>
</tr>
<tr>
<td>Streaming current</td>
<td>± 2 mA ± (0.2 % + 1 pA)</td>
</tr>
<tr>
<td>Cell resistance</td>
<td>5 Ω ... 20 MΩ ± (2 % + 0.5 Ω)</td>
</tr>
<tr>
<td>Differential pressure</td>
<td>SurPASS 3:</td>
</tr>
<tr>
<td></td>
<td>3500 mbar ± (0.2 % + 0.5 mbar)</td>
</tr>
<tr>
<td></td>
<td>external pressure supply (compressed N₂) required for differential pressure &gt; 1500 mbar.</td>
</tr>
<tr>
<td></td>
<td>SurPASS 3 Eco:</td>
</tr>
<tr>
<td></td>
<td>1200 mbar ± (0.2 % + 0.5 mbar)</td>
</tr>
<tr>
<td>Conductivity</td>
<td>0.1 ... 2 x 10⁵ mS/m</td>
</tr>
<tr>
<td>pH measurement</td>
<td>pH 1 ... 14 ± 0.05</td>
</tr>
<tr>
<td>Temperature</td>
<td>5° ... 40 °C ± 1.5 °C</td>
</tr>
</tbody>
</table>

### Electrolyte solution (reasonable measuring range for Ag/AgCl electrodes)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH value</td>
<td>pH 2 ... 12</td>
</tr>
<tr>
<td>Conductivity</td>
<td>0.1 ... 1000 mS/m</td>
</tr>
<tr>
<td>Temperature</td>
<td>20 ... 40 °C</td>
</tr>
<tr>
<td>Water</td>
<td>ASTM I grade</td>
</tr>
</tbody>
</table>

### Electrolyte solution (reasonable measuring range for gold electrodes)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH value</td>
<td>pH 2 ... 14</td>
</tr>
<tr>
<td>Conductivity</td>
<td>of 0.1 ... 150 mS/m for fiber, powder and conductive samples</td>
</tr>
<tr>
<td>Conductivity</td>
<td>of 0.1 ... 700 mS/m for planar, inert and not conductive samples</td>
</tr>
<tr>
<td>Temperature</td>
<td>20 ... 40 °C</td>
</tr>
<tr>
<td>Water</td>
<td>ASTM I grade</td>
</tr>
</tbody>
</table>

### Mains supply

- 115/230 VAC, 50/60 Hz
- Power consumption: 200 VA
- Fuses: T4A H 250V

### Dimensions

- 600 x 432 x 245 mm (D x W x H)
- Footprint: 612 x 536 mm (D x W)
Appendix A: Technical Data

Clearance 360 mm
Weight
SurPASS 3: 26 kg
SurPASS 3 Eco: 24 kg

Environmental conditions
Ambient temperature 20 ... 40 °C
Humidity 10 ... 80 % r.h. not condensing
Overvoltage category II according to EN 61010-1
Pollution degree 2 according to EN 61010-1
Noise emission 30 dB

Sample size requirement
Clamping Cell min. 35 mm × 15 mm or min. 17 mm in diameter,
max. thickness 40 mm
Cylindrical Cell particle size > 25 µm
Adjustable Gap Cell 20 mm × 10 mm, max. thickness 2 mm
Adjustable Gap Cell for Disks 14 mm or 15 mm in diameter, max. thickness 2 mm
Measuring Cell for Ceramic Membrane 25 mm Diameter: 25.2 ± 0.3 mm; Length: 100 mm ± 4 mm
Diameter: 10 ± 0.1 mm; Length: 96 mm ± 4 mm
Measuring Cell for Ceramic Membrane 30 mm Diameter: 30.8 ± 0.3 mm; Length: 100 mm ± 4 mm
Diameter: 13.1 ± 0.1 mm; Length: 96 mm ± 4 mm

Computer specification
Operating system Microsoft Windows® 7 32 bit and 64 bit
Service Pack 1 or later
(Windows 7 Starter Edition is not supported)
Microsoft Windows® 8 32 bit and 64 bit
Microsoft Windows® Server 2008 / 2008 R2 32 bit and 64 bit
Service Pack 2 or higher for Windows Server 2008
Microsoft Windows® 10 32 bit and 64 bit
CPU Intel® Core™ i3
RAM 4 GB (depends on operating system)
Hard disk 20 GB free disk space
Video Screen resolution at least 1024 x 768 at True Color (32 bit)
Communication port 1 free USB port

The SurPASS 3 is an instrument for indoor use at an altitude of up to 2500 m.
Appendix B: Declaration of Conformity

UK Declaration of Conformity

EC Declaration of Conformity
Appendix C: Requirements for Nitrogen Supply

WARNING

Risk of injury
As there is a risk of suffocation when using nitrogen for measurement, ensure adequate ventilation of the room.

External nitrogen supply is required for high pressure application (> 1500 mbar), adsorption studies or purging the electrolyte solution to prevent carbon dioxide dissolution.

The following requirements need to be fulfilled:
• Use nitrogen 5.0 for any of these purposes.
• If nitrogen is supplied directly from a gas container, a double-stage gas valve is required to reduce the primary pressure.
• If nitrogen is supplied from an in-house gas supply, where the primary pressure is already reduced, a single-stage gas valve is required.
• The operating pressure, i.e. the pressure applied on the position "IN" of the pressure control unit, shall be set to 6 bar to ensure correct functioning of the pressure control unit.
• The maximum operating pressure must not exceed 10 bar.
• The outlet port of the external gas supply should accommodate a pressure hose with 6 mm (1/4 inch).

NOTICE

The maximum pressure applicable to the instrument is 3500 mbar (high-pressure application).

Fig. C-1: Air Liquide pressure-reducing valve BS 20-1-2 with a resolution of 0.05 bar for use with a nitrogen supply with 20 bar primary pressure.

NOTICE

If using an inert gas instead of Nitrogen e.g. argon, make sure that the quality of the gas is 5.0.

The gas consumption of SurPASS 3 during operation is as follows:
• 0.2 l/min for purging the electrolyte solution
• 0.3 l/min for rinse and measurement cycles.